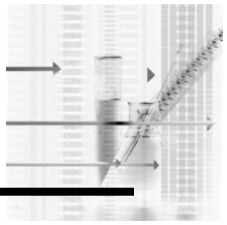
NICOMP 380 ZLS



User Manual

Particle Sizing Systems, Inc.

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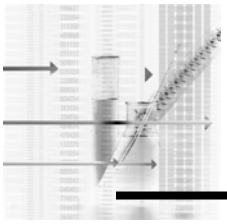

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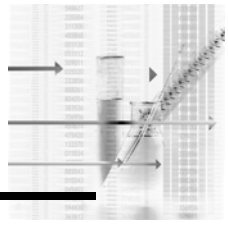
General Information



Registration

Please register your software by taking a moment to fill out the registration page provided. In keeping with our promise, we can easily provide two years of free software upgrades.

Just call us if you need information about our other products, or information about upgrading your existing system.

Technical Support

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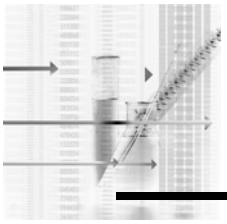


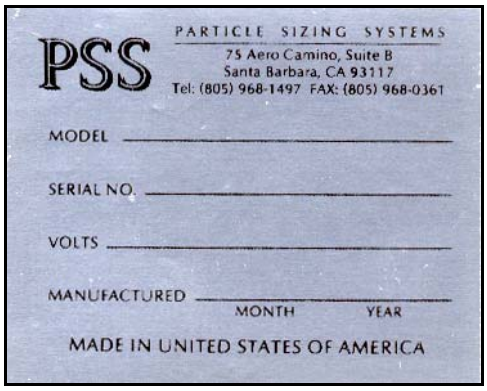
General Information

Safety Considerations

The NICOMP™ (and Autodiluter) Submicron Particle Sizer, is certified to conform to the applicable requirements of 21 CFR Subchapter J, 1040.10 and 1040.11 (Radiation Control for Health and Safety Act of 1968, 42 U.S.C 263f).

As presently constructed, this instrument is designated by the Bureau of Radiological Health Class I product. Exposure to negligible levels of *Laser Radiation* during normal operation results. The two labels below are affixed to the back panel of the Nicomp 380/Autodilute. They attest to the above *Safety Certification* and also establish the place and date of manufacture of the unit.

THIS EQUIPMENT CONFORMS
TO PROVISIONS OF
US 21 CFR 1040.10
AND 1040.11



Important: Read carefully before attempting to operate the Nicomp

If the Nicomp is to be used with the Autodilution option, then all liquid samples will be introduced into the system by means of a syringe or tube connected to the manual sampling valve that is located on the front panel of the instrument. In this case, NO entry into the sample holder space will be required.

Alternatively, if the Nicomp is to be used without the autodilution option, then all liquid samples will be introduced into the light scattering cell using 6 mm disposable glass culture tubes or standard 1-cm cuvettes. In this case, entry into the sample cell holder space will be required.

Access to the sample cell holder, necessary for inserting or removing a sample cell, is provided by a square opening at the front left corner of the top cover of the instrument. A rectangular dust cover with handle and three thumb screws are provided to keep the scattering cell and internal optical components free of excessive amounts of dust when the unit is not in use for extended periods of time and to prevent the laser light from scattering outside the unit during operation. During normal operation this cover can be secured with one screw and swung to one side to provide easy access to the cell holder. It can be swung shut during operation to keep out stray room light and keep in beam light being scattered by the particles.

During operation of the NICOMP™ Autodilute Submicron Particle Sizer, the *Top Cover* of the unit *Must Remain Closed* -- i.e. attached to the cabinet by means of the 3 screws provided. The *Warning* label on the cover warns of the possible exposure to the laser beam (a minimum of 5 milliwatts, 632.8 nm wavelength) if the top cover is removed for any reason while power is applied to the unit.

> **Important:** Any attempt to remove the front panel while the instrument is in operation may result in possible **Direct Exposure to Dangerous Laser Radiation**. Also, power must be off to the unit if the Autodilution cell is being replaced by the drop-in cell.

CE MARK

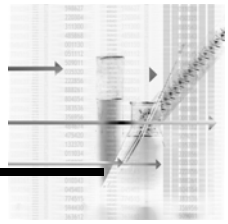
The **CE mark** (officially CE marking) is a mandatory marking on certain products, which is required if they are placed on the market in the European Economic Area (EEA). By affixing the CE marking, the manufacturer, or his representative, or the importer assures that that the item meets all the essential requirements of all applicable EU directives.

The CE mark is a mandatory European marking for certain product groups to indicate conformity with the essential health and safety requirements set out in European Directives. To permit the use of a CE mark on a product, proof that the item meets the relevant requirements must be documented. This has been achieved using an external test house which evaluates our particle size analyzers and its documentation. CE originally stood for *Communauté Européenne* or *Conformité Européenne*, French for European Conformity.

The following label is affixed to the back panel of the AccuSizer SIS to indicate that the instrument has passed CE mark testing and conforms to the European Union Directives for Electromagnetic Compatibility (EU EMC).

CE

ZETA POTENTIAL THEORY



INTRODUCTION

There has been increasing interest recently in techniques that provide a quantitative measure of the charge on colloidal particles in liquid suspension. Electrophoretic light scattering (ELS), which can measure the "zeta potential" of these particles, is one such technique. It can be implemented immediately following production of colloidal particles, as a means of estimating their stability against subsequent aggregation. Zeta potential analysis by ELS requires no special expertise; it can be performed as effectively in demanding process environments as in traditional laboratory settings. Fortunately, zeta potential analysis by ELS dovetails nicely in a technological sense with that of particle size analysis by dynamic light scattering (DLS).

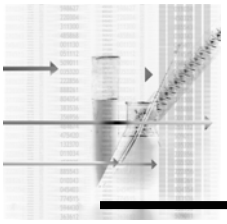
Given the ability of an ELS instrument to determine the zeta potential of colloidal particles in a simple and fast way, one is no longer limited to predicting the future stability of a colloidal system simply from its particle size distribution (PSD). The latter provides, at best, only a single "snapshot" of the present state of aggregation of the system. Such an isolated measurement of the PSD is unable to reliably predict the future rate of aggregation of the particles. For this purpose, a measurement of the zeta potential is invaluable.

ELECTROSTATIC REPULSIONS AND COLLOIDAL STABILITY

The physical mechanism that is used to stabilize most aqueous colloidal systems is electrostatic repulsion. The colloidal particles of interest are charged, resulting in their mutual repulsion at extended distances. Ideally, the repulsive forces are sufficiently strong to prevent the particles from diffusing close to each other, where short-range Van der Waals attractive forces dominate and lead to aggregation.

There are several means by which a net electrical charge can be attached to the surface of a colloidal particle. For particles that are normally uncharged, such as oil droplets in a homogenized aqueous suspension, charged molecules can usually be adsorbed onto the particle surface. This is most commonly accomplished using an ionic surfactant, consisting of a polar head group attached to a hydrocarbon "tail". The latter, being hydrophobic, associates with the interior of the uncharged particle, while the polar head group resides at the surface, in contact with the surrounding water, and dissociates, thereby imparting a net charge to the oil droplet, polystyrene bead, etc.

In other cases, the colloidal particles may already carry specific groups that are covalently bound to their surfaces and are ionizable. They carry a net positive or negative charge, or are neutral, depending on the pH of the surrounding aqueous solvent. Examples include many well known oxides, such as silica, alumina, titanium dioxide, etc. For these materials the extent and sign of the surface charge depends on the pH of the solution, as well as the pKs of the ionizable groups bound to the particle



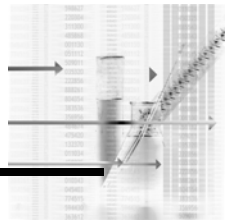
surface. Hence, the pH of the suspension will strongly influence the net charge of the colloidal particles and therefore their stability against aggregation.

The well-known DLVO theory of colloidal stability provides a reliable analytical framework for estimating the extent of repulsion or attraction between two particles in suspension as a function of their separation. The repulsive contribution to the interparticle "pair potential" depends on the particle charge and size and the concentration of mobile ions. The attractive portion depends on the strength of the Van der Waals attractive forces, as characterized by the Hamaker coefficient.

The charged mobile ions in solution distribute themselves between the large particles according to the laws of electrostatics and thermodynamics. The ions that carry a charge opposite to that of the colloidal particle surface are attracted preferentially to it. Consequently, the electrical potential in the solution, Ψ, produced by the charged particle decreases with increasing distance from its surface, due to the "screening" of its electric field by the mobile ions. The maximum value of the 7potential, Ψ_o, occurs at the surface of the particle. The higher the overall salt concentration, the steeper the "decay" in Ψ with distance from the surface. The charged particle surface and the diffuse layer of mostly oppositely charged ions surrounding it comprise the electrical "double layer". Its thickness is defined as the distance from the particle surface at which the electrical potential Ψ falls to 1/e of Ψ_o and is commonly referred to as the Debye-Hückel screening length, κ^{-1}. For a monovalent salt concentration of 1M, length κ^{-1} is only 3nm, while for 0.01M this length increases to 30 nm.

The zeta potential, ξ, also called the electro kinetic potential, is defined as the value of the electrical potential at the "shear plane" of the particle. For typical colloids this point is close to the actual surface of the particle. For relatively low concentrations of added salt, the zeta potential as measured by ELS should provide a good representation of Ψ_o at the surface of the particles.

In summary, if the electrical double layers of a colloidal system overlap, i.e. if κ^{-1} exceeds the average interparticle separation, then the system will usually be stable, given a moderate amount of charge on the particles. However, if the concentration of salt ions is high enough to lead to significant shrinkage of the electrical double layers, so that they no longer overlap, then the value of ξ will be important in establishing whether the repulsive electrostatic potential barrier between neighboring particles is high enough to preclude their agglomeration due to short-range attractive Van der Waals forces.



REVIEW OF ELS: MOBILITY, DOPPLER SHIFT AND ZETA POTENTIAL

As its name implies, the ELS technique is based on the scattering of light from particles that move in liquid under the influence of an applied electric field. The charged particles quickly reach a constant "terminal" velocity υ, proportional to the magnitude of the field, E. The proportionality constant, μ, defines the electrophoretic mobility,

$$\nu = \mu E \qquad (1)$$

The units of electric field are V/cm, and therefore the units of μ are cm/s/V/cm (cm^2/Vsec). Because typical velocities are so small, it is useful to express μ in terms of the "mobility unit", M.U. (μm/s/V/cm) which equals 10^{-4} cm/s/V/cm.

The moving particles scatter the incident light at a frequency ν_s, which is Doppler shifted with respect to the incident frequency ν_o. The extent (and sign) of the Doppler shift in frequency, $\Delta\nu$, depends on the velocity of the particle, the wavelength of the incident light beam (in the liquid medium) and the angle or scattering. The relationship between these quantities is given simply by

$$2\pi\,\Delta\nu = \mathbf{K} \bullet \mathbf{v} \qquad (2)$$

where **K** is the scattering wave vector, familiar from the theory of DLS. The magnitude of K is given by

$$K = (4\pi n/\lambda_o) \sin \theta/2 \qquad (3)$$

where λ_o is the wavelength of the incident light beam (in vacuum), n the refractive index of the solvent, and θ the angle at which the scattered light is detected. The relationship between the electric field and particle velocity vectors, the incident wave vector k_o and the scattered wave vector k_s is summarized in Figure 1.

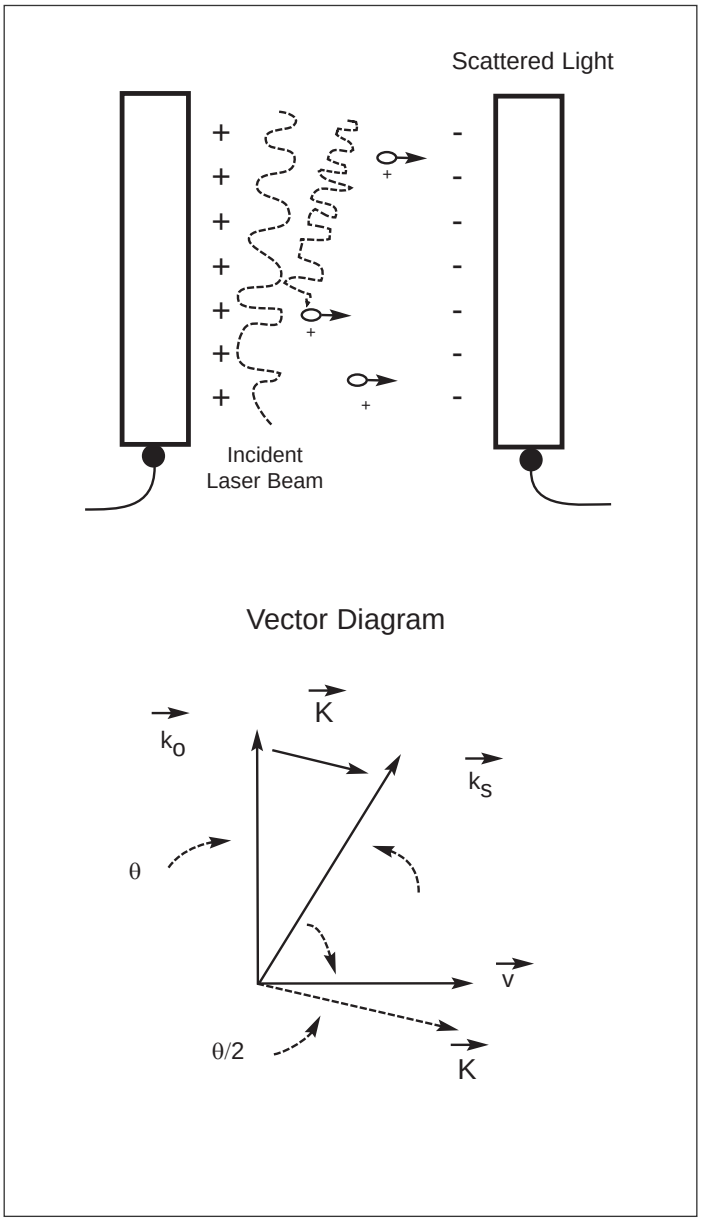
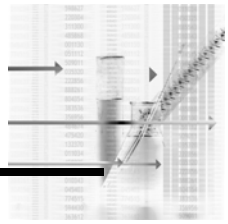


Figure 1: Relationship between the incident laser beam and scattered light wave for ELS; vector diagram relating these to the scattering wave vector K and particle velocity v.



The Doppler shift, $\Delta\nu$, in the frequency of the scattered light is easily computed from Figure 1 and Eq'ns 2 and 3,

$$\Delta\nu = (K\, v/2\pi) \cos \theta/2$$
$$= (2nv/\lambda_o) \sin \theta/2 \cos \theta/2 \qquad (4a)$$

Use of a common trigonometric identity reduces this to,

$$\Delta\upsilon = (nv/\lambda_o) \sin \theta \qquad (4b)$$

Finally, substitution of Eq'n 1 into the above equation allows the electrophoretic mobility, μ, to be computed from the measured Doppler shift, $\Delta\nu$, for a given applied electric field strength, E,

$$\mu = (\lambda_0 /n \sin \theta)(1/E)\, \Delta\nu \qquad (5)$$

It is useful to attach some typical numbers to the above formula. For the instrument recently developed by the authors, the NICOMP™ 380 ZLS Particle Size/Zeta Potential Analyzer, the relevant parameters for Eq'n 5 are: λ_0 = 0.6328 μm (HeNe laser), n = 1.33 (water) and θ = 14.8 degrees. In this case, Eq'n 5 reduces to μ (M.U.) = 1.867 (1/E) $\Delta\nu$.

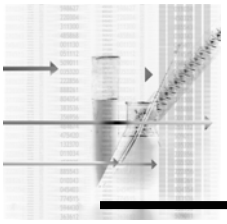
Finally, the value of the mean zeta potential, ξ, is obtained from the electrophoretic mobility, μ. If the concentration of mobile ions in solution is sufficiently high that the thickness of the electrical double layer, κ^{-1} , is small compared to the mean diameter of the particles, a (i.e. $\kappa a >> 1$), then the Smoluchowski approximation applies. In this case, ξ is related in a simple, linear way to μ,

$$\xi = \eta\mu/\varepsilon \qquad (6)$$

where η is the viscosity and ε the dielectric constant of the solvent.

In the opposite limit of ion screening, known as the Hückel limit, where $\kappa a << 1$, the electrical double layers of neighboring particles overlap significantly. In this case the right-hand side of Eq'n 6 is multiplied by 3/2. For intermediate values of κa, an analytical approximation is needed in order to estimate the value of ξ from μ.

As expected (Eq'n 4b), the magnitude of the Doppler shift is proportional to the component of the velocity vector lying along the direction of detected scattered light, k_s, proportional to sin θ. Hence, it would appear that the measured Doppler shift, $\Delta\nu$, can be increased simply by increasing the angle, θ, of detection of the scattered light. However, in practice, this is the opposite of what is required to optimize an ELS measurement, for reasons that are both practical and theoretical.



The practical requirement for small angles is related to the geometrical constraints imposed by the electrodes immersed in the sample solution. The parallel electrodes must be relatively closely spaced (typ. 2-4 mm), in order to achieve a uniform electric field where the scattered light is collected and also to reduce the applied voltage needed to achieve significant values of field strength. (The lower voltage reduces the amount of Joule heating and resulting convection that can occur at moderately high salt concentrations.) Hence, there is only a relatively small range of angles at which the scattered light can be collected (i.e. not blocked by the electrodes].

However, there is a much more important, theoretical consideration which dictates the use of a small scattering angle. In addition to being subjected to a constant drift velocity due to imposition of an electric field, the particles always experience random-walk, Brownian motion, or diffusion, due to random collisions of the surrounding solvent molecules. Indeed, this is the ever-present phenomenon that gives rise to fluctuations in the scattered light intensity which are analyzed by the DLS technique. The autocorrelation function of these fluctuations yields the diffusion coefficient of the particles, from which the particle diameter can be derived.

The random motions of the particles due to diffusion also give rise to fluctuating Doppler shifts in the frequency of the light waves scattered by each particle. These random shifts are superimposed on the constant shift $\Delta\nu$ associated with the drift velocity, ν, caused by the applied electric field.

In the "heterodyne" light scattering technique which we use for the ELS measurement (described below), the half width, Γ, of frequency broadening of the detected scattered light due to Doppler shifting by the diffusing particles is given by

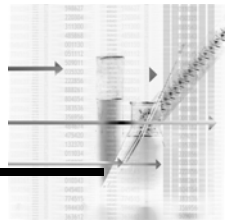
$$\Gamma = D K^2 \qquad (7)$$

where K is the same scattering wave vector used above (Eq'n 3) and D is the diffusion coefficient of the particles, given by the Stokes-Einstein relation,

$$D = kT/3\pi\eta a \qquad (8)$$

where k is Boltzmann's constant and a is the particle diameter. The Doppler shift due to electrophoretic mobility decreases with decreasing scattering angle, approximately as $\sin \theta/2$ (Eq'n 4a), while the frequency broadening due to diffusion also decreases with angle, but as the square of $\sin \theta/2$. Hence, the ability of the ELS technique to measure small frequency shifts associated with low electrophoretic mobilities dictates operation at a relatively small angle.

It is instructive to compare the extent of frequency broadening, Γ, of the scattered light due to random diffusion with the typical range of Doppler shifts $\Delta\nu$ encountered from



electrophoretic mobility, typically 1-100 Hz. We compute the value of Γ for two different scattering angles: 90°, used typically for DLS particle size analysis, and 14.8°, which we use for our zeta potential measurements. We assume HeNe laser light, water and a temperature of 23°C.

The value of Γ is inversely proportional to the particle diameter. For a = 1000nm, Γ = 26.9 Hz at θ = 90° but only 0.89 Hz at θ = 14.8°. For a = 100nm, the respective values are 269Hz and 8.9Hz, while for a = 10nm, the corresponding values are 2690Hz and 89.3Hz.

INSTRUMENT DESIGN: CAPABILITY FOR BOTH ELS AND DLS

A simplified block diagram of our combined particle size and zeta potential analyzer, the NICOMP 380 ZLS, is shown in Figure 2. The instrument employs a novel design, which permits both multi-angle particle size analysis by DLS and low-angle zeta potential analysis by ELS, using a minimum number of optical components. A single, precision optical fiber/collimator, together with a high-resolution stepper motor (0.9°/step), is used to implement both the ELS and DLS measurements. This design offers scientific flexibility, without sacrificing ease of use and reliability.

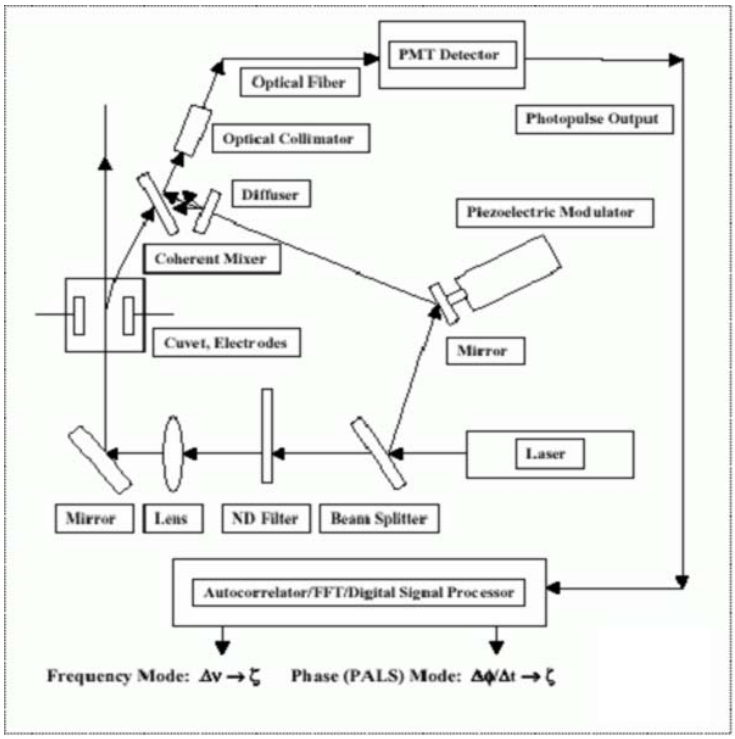
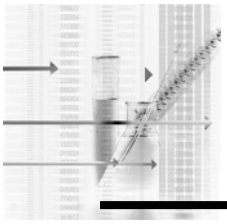


Figure 2: Simplified schematic diagram of the NICOMP 380 ZLS Zeta Potential and Particle Size Analyzer, based on ELS and DLS.



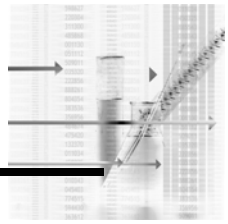
In the ELS mode, the optical fiber (OF) is rotated to an external angle of 19.8°, which translates (for water) into a scattering angle of 14.8°. Scattered light (ELS) at this angle is collected by OF and transmitted to the PMT detector. However, in addition a small fraction (approx. 4%) reflected by of the original laser light beam, referred to as the "local oscillator" (LO), is split off by beam splitter BS1, mirror M2 and directed at a second beam splitter, BS2. A small fraction of the LO light wave is reflected by BS2 into the OF pickup, where it mixes coherently with the LS wave. The resulting coherent superposition of light signals is transmitted to the PMT detector by the optical fiber. By analogy to a radio receiver this scheme is referred to as a "heterodyne" light scattering system.

Accurate heterodyne detection requires that the intensity of the LO wave greatly exceed (i.e. 20:1 to 30:1) that of the LS wave with which it is mixed. A variable neutral-density filter (NDF) is used to automatically adjust the intensity of the detected ELS wave relative to the intensity of the LO wave. Typical intensities (expressed as photopulse rates) for the two waves are 2,000-4,000 kHz and 100-200 kHz, respectively.

The frequency of the LO wave, ν_o, is enormous (e.g. 5×10^{14} Hz for red HeNe light) compared to the small Doppler shift, $\Delta\nu$, expected in the frequency of the ELS wave. Hence, it is not feasible to measure directly this miniscule relative change in frequency. However, the coherent mixing of the two waves at the detector provides the desired result. The electronic (photopulse) signal produced by the PMT detector contains a component which effectively oscillates at the "beat note", or difference, frequency between the two individual optical frequencies.

All that is required to determine $\Delta\nu$ is to measure the frequency power spectrum of the PMT output signal. In practice, however, the overall accuracy, or signal-noise ratio, of the measurement can be greatly improved by "shifting" the frequency range of interest from zero Hz ("DC") to some convenient frequency, away from the influence of common sources of electronic noise, and drift, which are especially prevalent at very low frequencies. Hence, it is useful to "add" an arbitrary, fixed frequency to the existing frequency of the LO wave.

This added shift is conveniently accomplished by using the Doppler effect once again. A piezoelectric translator, PZT, attached to mirror M2, is driven at some arbitrary frequency, ν_{PZT}-- 260 Hz in our case. The frequency of the LO wave is shifted upward to $\nu_o + \nu_{PZT}$. The "beat note" which therefore appears on the output of the PMT detector, representing the difference in frequencies of the ELS and LO waves, is similarly translated upward, to $\nu_{PZT} + \Delta\nu$. The electrophoretic mobility will therefore be manifested as a frequency shift $\Delta\upsilon$ with respect to υ_{PZT}. Proper design and synchronization of the electronic waveforms that drive the cell electrode voltage and PZT device allow the sign, as well as the magnitude, of the frequency shift to be determined.



The PMT photocurrent signal, consisting of individual photopulses of average frequency 2,000-4,000 kHz, is passed to a multichannel digital autocorrelator (AC). In the case of our NICOMP 380 ZLS instrument, the AC uses four fast digital signal processors (DSPs) with 32-bit architecture, operating with an adjustable number of channels and channel width. Typical ELS measurements are made using either 256 or 512 channels, with 500 μs/channel. The desired frequency shift of the ELS wave relative to the LO wave is obtained by Fourier analysis of the autocorrelation function (ACF) of the PMT photopulse signal. This is accomplished using a fast Fourier transform (FFT) algorithm, which yields the power spectrum (PS) of the heterodyne signal, ELS + LO.

Finally, the electrode assembly can be removed from the disposable sample cuvet, and the system used to determine the particle size distribution (PSD) by dynamic light scattering (DLS). It in possible to use a modified version of the heterodyne scheme discussed above for zeta potential analysis. In this case, the PZT modulator must be turned off, so that the LO wave is no longer Doppler shifted in frequency.

However, for technical reasons it is preferable to make the DLS measurement using the more conventional homodyne scattering approach, in which the LO wave is turned off and only the light scattered from the diffusing particles is detected.

In the ideal case of uniform-size particles, the ACF is a simple decaying exponential function with decay time τ, where $1/\tau = 2\Gamma$, and Γ is given by Eq'n 7. The particle diameter, a, is then obtained from Eq'n 8. For non-trivial distributions, including simple log-normals and more complex bimodals or skewed unimodals, the PSD can be estimated from the ACF using either cumulants analysis or a more sophisticated Laplace transform algorithm. The multiangle capability shown in Figure 2 is invaluable for broad PSDs at larger diameters (a > 200nm), when there is significant angular dependence of the scattering intensity due to intraparticle Mie scattering.

REPRESENTATIVE ELS RESULTS

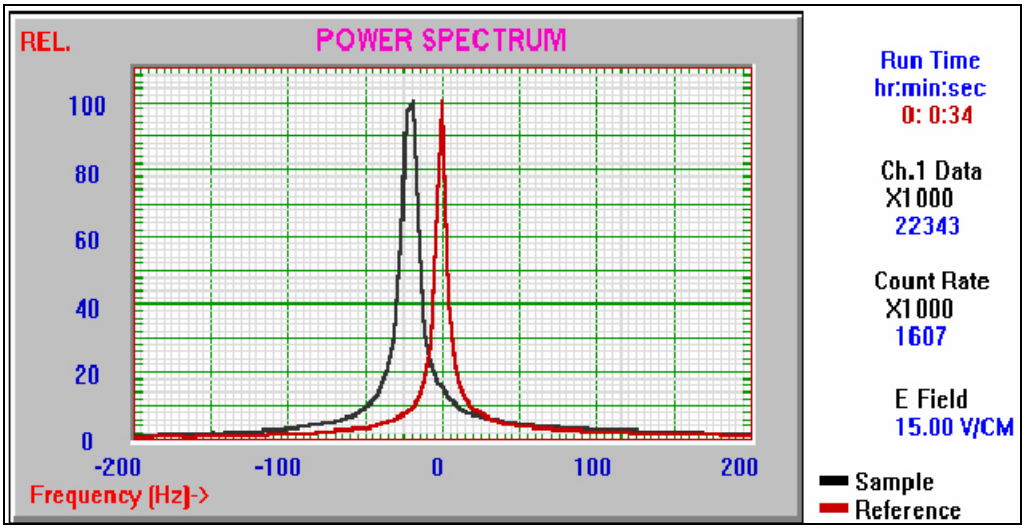


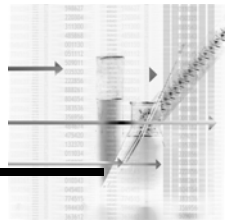
Figure 3: ELS power spectrum obtained for 262-nm polystyrene latex spheres
E = 10 V/cm.

Figure 3 shows the ELS power spectrum (PS) obtained for a dilute aqueous suspension (5000:1 of 10% stock) of polystyrene latex spheres of diameter 262nm, with 0.2% added surfactant (SDS) to ensure full charging of the spheres. The peak at the center is the "reference" PS, with zero applied E-field; the left-most (ELS) peak was obtained with an applied field of 10V/cm.

The frequency scale has been offset by the modulation frequency of the LO wave, ν_{PZT} = 260Hz, which shifts the reference peak to zero Hz. The center of the ELS peak is shifted by -19.3Hz with respect to the reference peak, corresponding to an electrophoretic mobility μ of -3.61 M.U., or a zeta potential ξ of -48.8mV, assuming the Smoluchowski limit. The ELS peak is broadened significantly, indicating a range of mobilities, or zeta potentials, rather than a single value.

Figure 4 shows the ACF from which the shifted ELS peak in Figure 3 was obtained by FFT. This measurement was made using 256 channels and a channel width of 500μs. As expected, the ACF resembles a smoothly decaying oscillating function, with a frequency corresponding to the mean frequency shift of the ELS wave relative to the LO wave -- in this case, 260-19.3=240.7Hz.

The characteristic decay time of the "envelope" of the oscillatory function is inversely proportional to the width of the ELS peak. This width depends on the range of ξ values,



as well as normal diffusion broadening (Eq'n 7), inversely related to the mean particle diameter (Eq'n 8).

The broadening due to diffusion alone can be inferred from the width of the unshifted reference peak.

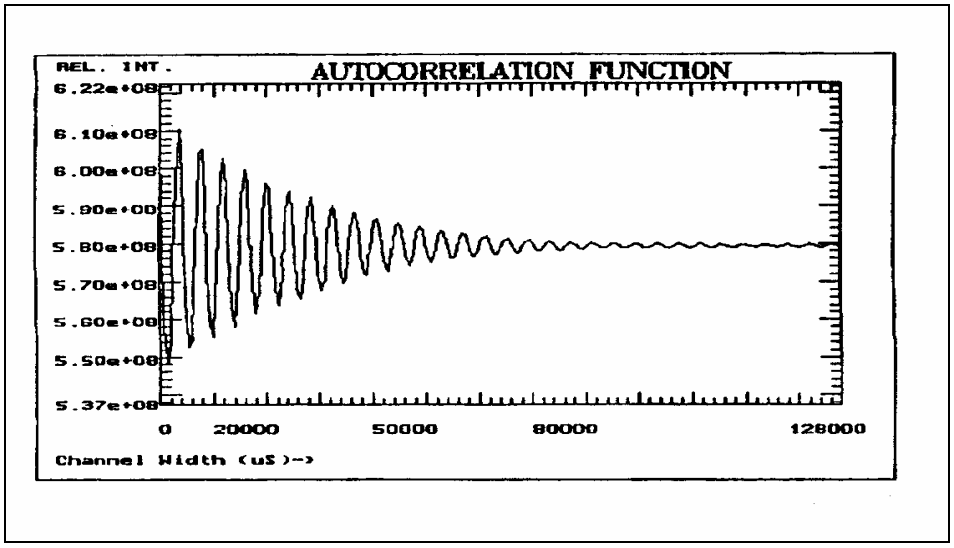
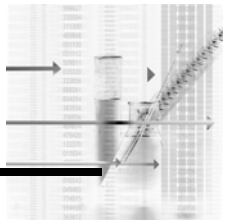


Figure 4: Heterodyne autocorrelation function (256 channels, 500µs/ch.) from which the ELS power spectrum (Figure 3) was obtained by FFT.

Interestingly, the zeta potential for these latex particles is observed to decrease when the amount of added SDS surfactant is reduced. The latex beads are stabilized during production with a substantial negative charge imparted by an adsorbed layer of anionic surfactant. However, if this concentrated dispersion is highly diluted, some of the surfactant must leave the particle surfaces in order to maintain the low concentration of free surfactant monomers and micelles in the aqueous phase. Hence, additional anionic surfactant must be added to keep the particles sufficiently charged to avoid aggregation.

In summary, it is evident that the technique of ELS constitutes a powerful tool for the measurement of electrophoretic mobilities and zeta potentials of charged colloidal systems. It is especially useful when combined with DLS technology for multi-angle, high-resolution particle size analysis in the same instrument package.



INITIAL HARDWARE INSTALLATION

1. Connect the 25-pin male connector of the cable provided to the port identified on the back of the Nicomp instrument.

2. Connect the 9-pin female end of the connector to a serial port on the PC controller.

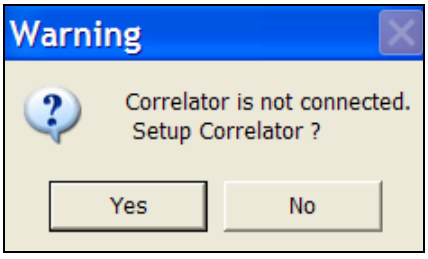
3. Plug the power cable provided into the back of the unit.
4. If the unit has the Autodilution feature, place the drain line into a waste bucket.
5. Apply power to the PC controller.
6. Install the Nicomp software package. Review the Software section of this manual.
7. Apply power to the Nicomp.

INITIALIZING

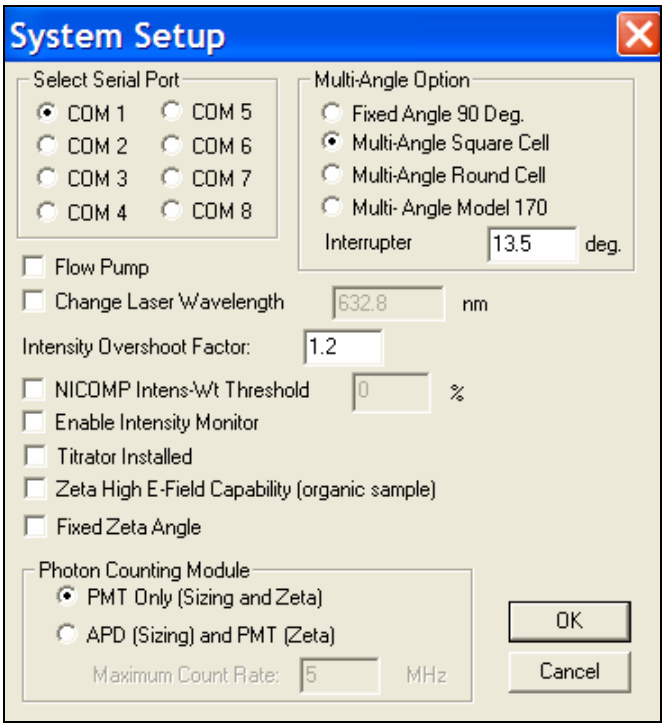
Following is the procedure for initializing the Neutral Density (ND) filter in the 380/ZLS instrument for both zeta potential and particle sizing modes of operation.

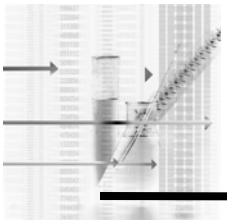
1. Power up the PC.

2. Click on the **ZPW388** icon to access the software with no power applied to the 380/ZLS unit. The following window displays:



3. Position the highlight bar over the **Setup** option of the **Tool Bar** and click once. The following screen displays:





4. Select the correct serial port. In most cases this will be **Serial Port Com 1**.

5. Click on the **Multi-Angle Square Cell** option.

6. Click on **OK**. The system defaults to the Particle Sizing operating mode.

7. Remove the top cover of the 380/ZLS unit and remove any Velcro or elastic bands that restrain the moveable arm holding the optical fiber. DO NOT apply power to the 380/ZLS unit until the arm has been unrestrained and is free to rotate (stepper motor shaft). It is important to note that the arm MUST be restrained again prior to shipping the unit. In order to avoid damaging the optical fiber and/or losing the optical alignment that was set up at the factory.

8. Prepare a dilute (slightly turbid) aqueous suspension of submicron polystyrene latex (suggestion: 265-nm) by mixing 1 drop of latex with DI water and pipette some of it into one of the square plastic cuvets which are supplied with the unit.

9. Fill the cuvet to about 2/3 capacity. The laser beam will enter the cuvet ¾" above the (outside) bottom.

10. Place the sample cuvet into the sample holder. It may be necessary to remove the drop in block or flow thru cell to accommodate this.

11. Apply power to the 380/ZLS unit. The moveable arm should automatically step toward the smaller angles until it reaches the reference interrupter, at which point it should reverse direction and commence stepping toward larger angles. If the ZPW388 software is set to Particle Sizing mode, the arm will come to rest at the default scattering angle for this mode, 90 degrees.

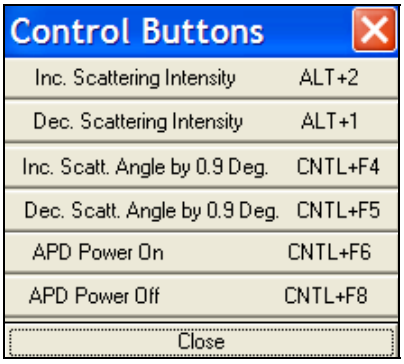
12. Press ALT-CTRL-I simultaneously to initialize the neutral density filter. The variable neutral density (ND) filter should be observed to rotate automatically toward progressively lower amounts of filtering, thereby allowing the intensity of the laser beam which passes through the filter to become progressively brighter. The firmware system will then detect the "edge" of the ND filter, where the filtering reaches a maximum ND = 3.0 units, where the attenuation of the beam is greatest, approximately 1000:1. The ND filter will then advance slightly further and stop, so that the attenuation is approximately maximum. The software designates this as ND position = 0. The neutral density filter can be moved a total of 255 increments after initialization ND = 3 is at position zero and ND = 0 is at position 255.

    From this automatic sequence, the system will have found and stored in memory the location of the "edge" of the ND filter, so that all future positions of the filter can be remembered relative to that reference location. It should be noted that this ND filter reference location will be stored in the configuration file, ZPW388.CFG, which will be

created in the same directory as ZW380.EXE when one exits from the program. The firmware should then run through the initialization procedure automatically, as indicated above. The initialization procedure can always be initiated manually by pressing ALT+CTRL + I simultaneously.

Note that the intensity which arises from the test sample. It should be very low, since the ND filter is positioned for nearly maximum light beam attenuation. The filter must therefore be rotated so that the desired light scattering intensity for sizing is achieved – typically 300-400 kHz is recommended for a sample that scatters adequately.

13. Click on the **Particle Sizing** menu and choose Control Buttons. The following window displays:


Control Buttons
Inc. Scattering Intensity ALT+2
Dec. Scattering Intensity ALT+1
Inc. Scatt. Angle by 0.9 Deg. CNTL+F4
Dec. Scatt. Angle by 0.9 Deg. CNTL+F5
APD Power On CNTL+F6
APD Power Off CNTL+F8
Close

14. The ND filter can then be rotated in the desired direction by clicking repeatedly on **Increase** or **Decrease Scattering Intensity** until the desired intensity value of **120 kHz** is reached. This particular position of the ND filter will then be stored by the system, so that whenever the unit is returned to the **Particle Sizing** mode of operation, the ND filter wheel will return to that position.

Note: The preceding discussion presumes that there was no significant change in the "vertical alignment" of the moveable arm/optical fiber during shipping of the instrument. In general, this should be the case. Unfortunately, there is no obvious way for the user to determine whether the arm/fiber assembly has moved slightly, up or down, with respect to the line source of scattered light in the sample cuvet. Any gross misalignment of the arm/fiber is, of course, easy to determine from the fact that the level of scattered light intensity is below levels that are normally achieved and expected. It is perhaps a good idea to optimize the vertical alignment during the initial setup of the instrument, described in a separate section. However, this procedure should be carried out by a trained PSS service representative.

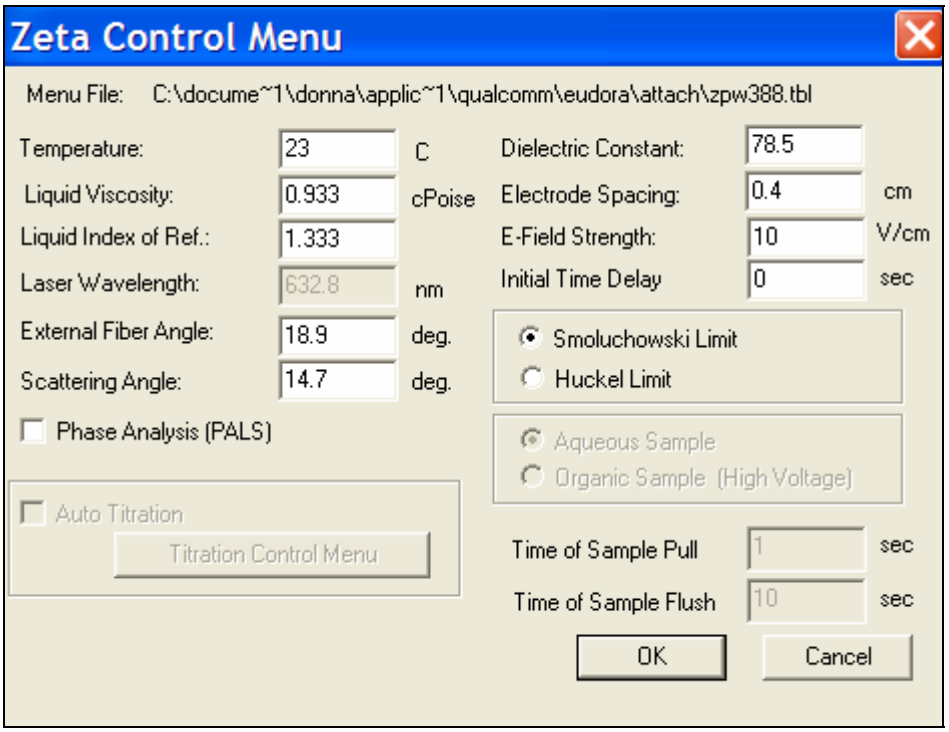
After the rotation of the ND filter has been adjusted to produce the desired, typical level of scattering intensity in the Particle Sizing mode of operation. Close the Control Buttons window, one is now ready to switch to the Zeta Potential mode of operation.

15. Position the highlight bar over the **Particle Sizing** option of the Tool Bar and click once. The particle sizing menu re-displays (see Screen 3).

16. Position the highlight bar over the **To Zeta Potential** option of the pull down menu and click once. Answer Yes to the window that asks to move to Zeta Potential measurement.

    After this choice has been made, the ND filter will rotate automatically to the location of maximum attenuation (position 0). The multi angle arm with the fiber optic cable will then automatically step toward smaller angles, make contact with the reference detector and then move to the default small angle that is required for the zeta potential mode of operation (18.9 degrees).

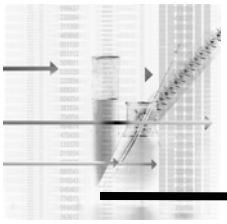
    After the arm has come to rest at a small angle, the **Zeta Control Menu** displays:



Note: *The default value of the angle should be 18.9 degrees in all cases. If the default is not 18.9 the user can click in the box and change the value to 19 (the system will correctly interpret to mean 18.9 degrees).*

17. Make all values as above and Click on **OK**. The angle for the zeta potential mode of operation will then change to the desired value of 18.9 degrees. This value will then be stored in memory and in the Control menu for future operation.

    It may be necessary to establish that the “local oscillator” (LO), or reference, light beam is properly aligned with respect to the optical fiber carried by the moveable arm.



Reference Beam

This reference beam is created by the clear silica beam splitter that lies in the path of the laser beam, before it enters the focusing lens/mirror assembly near the sample cell holder. The incident laser beam is partially reflected by the silica window, thereby creating a secondary beam with an intensity about 4% of the intensity of the main laser beam. This secondary beam impinges on a miniature mirror attached to a piezoelectric modulator (PEM, which vibrates at a frequency of approximately 260.4 Hz. This beam is then redirected toward an optical "mixer" (i.e. beam splitter), located in front of the moveable arm/optical assembly. A second clear silica window serves as the optical mixer, located between the source of the light scattering in the cell holder and the input of the optical fiber. It is oriented at a 45-degree angle with respect to the axis of the fiber, which is pointed toward the (approximate) center of the sample cuvet.

Scattered light originating from the sample is able to reach the input of the optical fiber with only 4% reflected away. The reference light beam coming from the PEM, on the other hand, can also be "mixed" with the scattered light signal as it enters the fiber by virtue of the fact that about 4% of the impinging reference beam will be reflected by the silica window into the optical fiber. The only remaining problem is the fact that proper alignment of the reference beam with respect to the scattered light signal at the fiber entrance is normally difficult to achieve. However, this alignment requirement is conveniently relaxed by using a "diffuser" to randomize the arriving reference beam. This is the reason why a small piece of frosted plastic has been mounted close to the optical mixer, in the path of the arriving reference beam.

One can see the reference beam when the cover is off the instrument when the "reference beam on" icon is clicked. The shutter that blocks the reference beam during normal sizing operation of the instrument will move and the PEM will start to vibrate. One can always ensure that the measurement is progressing by the sound this PEM makes.

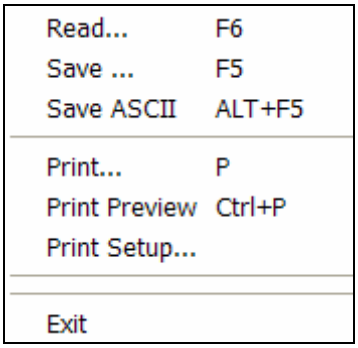
Upon installation of the instrument a PSS representative will optimize the alignment by carefully adjusting the thumbscrews located on the two-dimensional tilting stage, on which is mounted the PEM unit. There are not many reasons that the user will have to readjust this alignment. In any case, the tilting stage should be adjusted in order to produce a total detected intensity of approximately 2000-4000 kHz. Any value higher than 4000 kHz should be avoided by slightly "de-tuning" the tilt stage.

FILE

The following provides a brief summary of the File options that are used during the use of the Windows CW388 Software

Access of the File options:

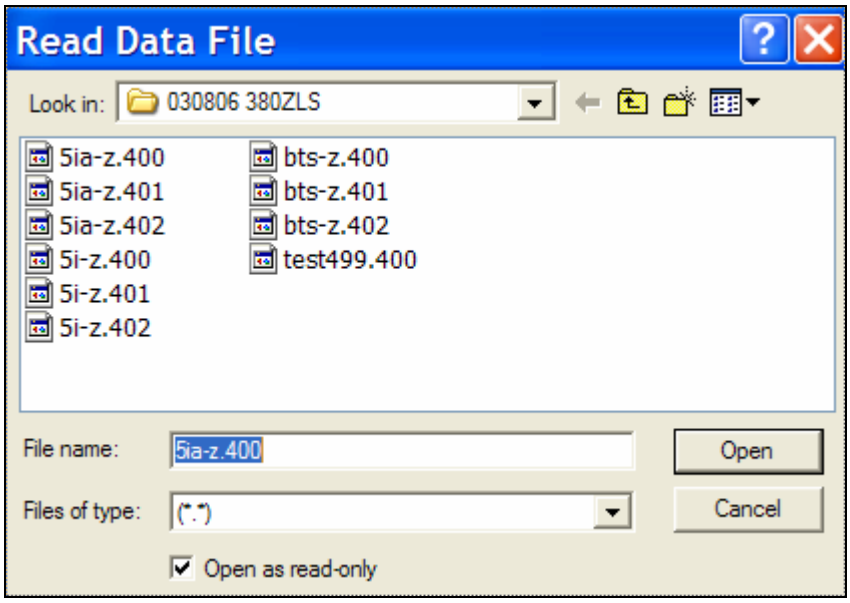
1. Position the highlight over the **FILE** option and click once using the mouse. The following window of File options displays:



2. Position the highlight bar over the desired selection and click once. Following is a description of each of the options offered.

Read

A data file that has been stored following a measurement can be retrieved to display the resulting particle size distribution (PSD), with the desired weighting. When this option is selected, a list of data files will display in the Read Data File window such as in the example below:



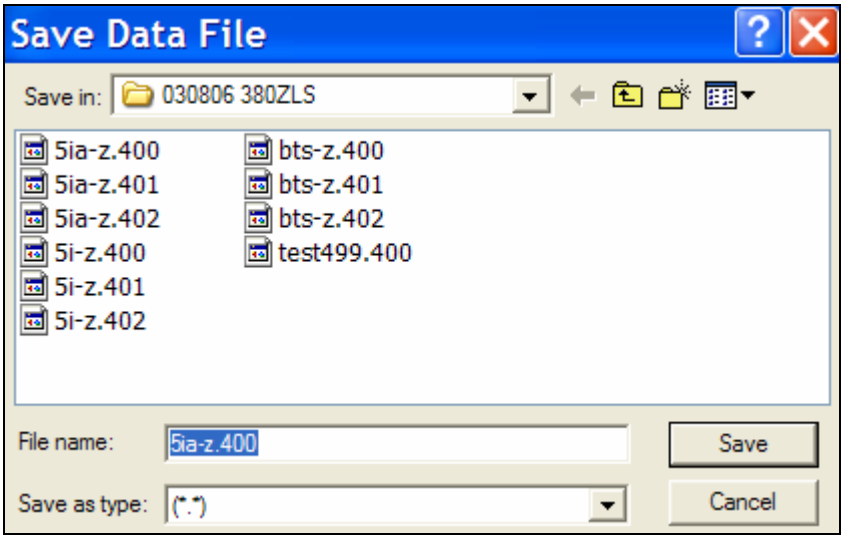
1. Position the highlight bar over the data file of interest and click the mouse once. The Zeta Potential History will display.

Save

This option is used throughout the ZPW388 software whenever a data file is to be saved. To access this option:

1. Position the highlight bar over the **File** option and click the mouse once.

2. Position the highlight bar over the **Save As** option and click the mouse once. The following screen will display:



Save ASCII

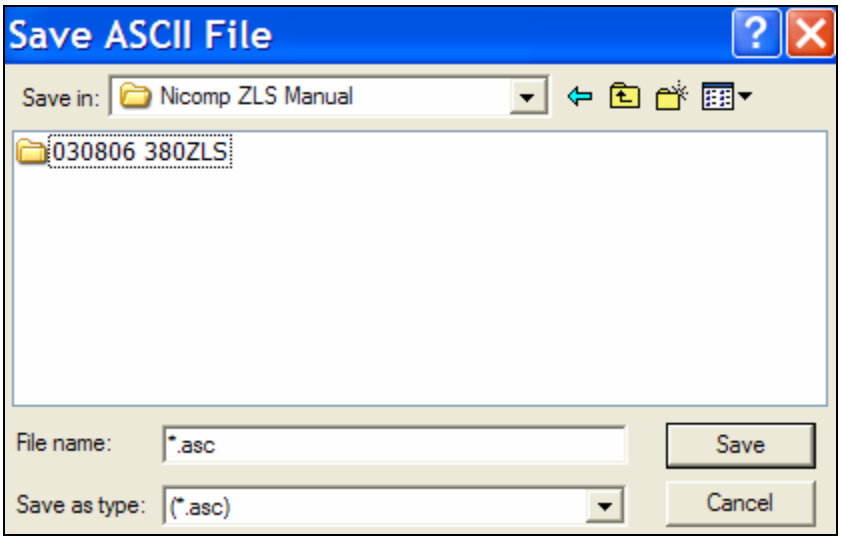
Use this option to save the data collected for a particular sample to an ASCII file format. The data can then be imported to a spreadsheet program for presentation.

To create new files in standard **ASCII** format to export data files into other software packages (e.g. spreadsheets), for manipulation of the original data follow these steps:

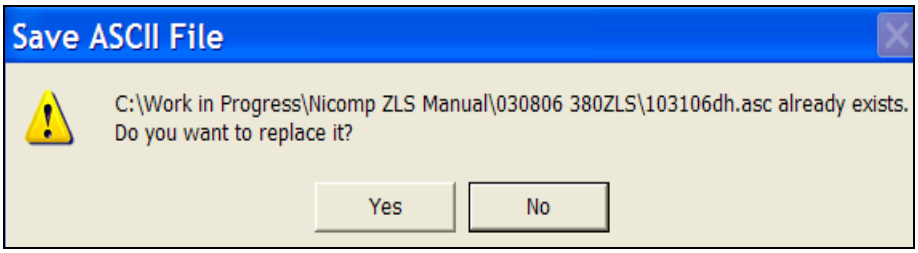
1. Select a data file.

2. Position the highlight bar over the **File** option and click once.

3. Position the highlight bar over the **Save ASCII File** option using the mouse and click once the following window displays:



1. Type in the desired file name. A new file in **ASCII** format will be created and stored in the **Data Directory** with the file extension **.asc**. If the same file name already exists, the following message will appear:



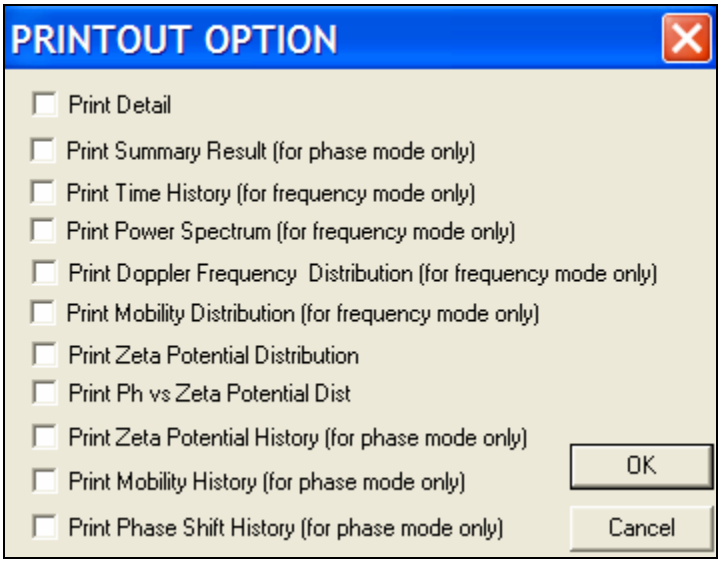
2. Click on:

   Yes: the existing **ASCII** file having the same file name will be erased, and the new one stored in its place.
   No: no new file will be stored.

Print

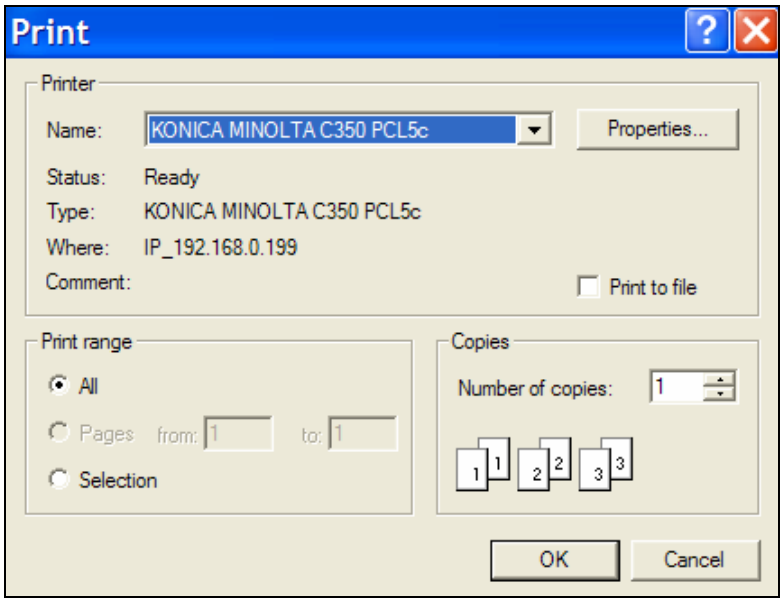
Printouts of the sample distributions that display on the computer monitor can be achieved using this option. Please refer to Appendix A for printout samples.

1. Click on the **File** Window option and position the highlight bar over the **Print** option and click. The following **Printout Option** window will display:


PRINTOUT OPTION
Print Detail
Print Summary Result (for phase mode only)
Print Time History (for frequency mode only)
Print Power Spectrum (for frequency mode only)
Print Doppler Frequency Distribution (for frequency mode only)
Print Mobility Distribution (for frequency mode only)
Print Zeta Potential Distribution
Print Ph vs Zeta Potential Dist
Print Zeta Potential History (for phase mode only)
Print Mobility History (for phase mode only)
Print Phase Shift History (for phase mode only)
OK
Cancel

2. Click on the square located to the left of the print selection. A black check mark will display in the box selected.

3. Click on **OK** to start printing the distributions and/or plots. The following Print window will display:



1. Verify the printer type selected. If changes need to be made follow these steps:

2. Click on the Down arrow. A window of all of the printer brands and types will display. Selection of the correct printer driver software depends on the setup of this option.

3. Position the highlight bar over the **Printer** type and model that is currently hooked up to the computer being used.

4. Click the mouse once.

5. Click on the **OK** button. The Print windows will re-display with the printer and type and model selected.

6. Position the cursor over the print range desired.

   All - will print all data pertinent to the distribution being reviewed.
   Pages - will print the range of pages desired.
   Selection - will only print those pages desired.

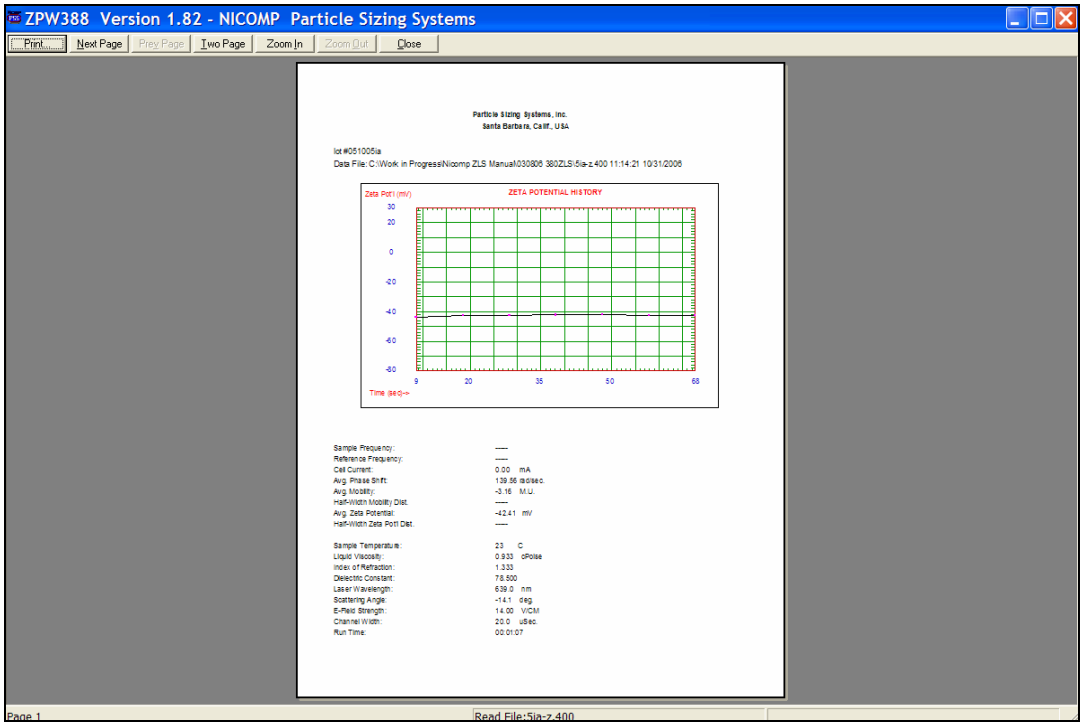
7. Position the cursor in the **Copies** option and type a number for the number of copied desired for the printout. The default is set to one.

8. Click on the **OK** button to start printing.

Print Preview

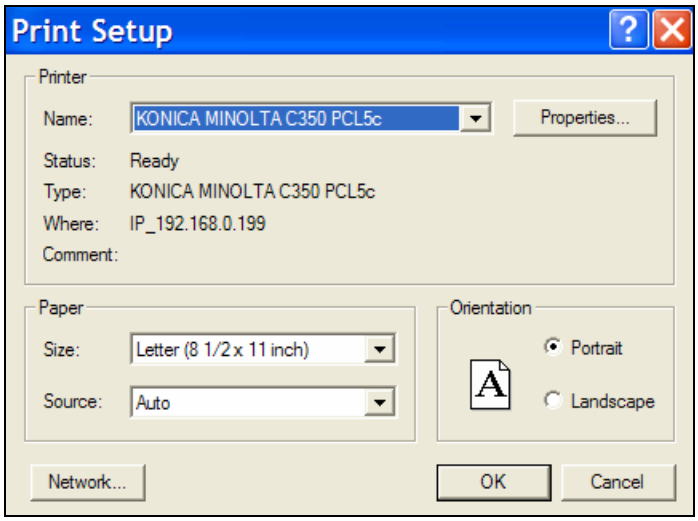
Allows the printout to be viewed prior to being sent to the printer. Following is an example of the window that will display. A data file must be accessed prior to using the Print Preview option.

Print - will print the distribution that is being previewed
Next Page - will advance to the next page of the preview
Two Pages - will preview two pages of the same file side by side
Zoom In - provides the ability to zoom into the distribution to examine the fine details of the distribution
Close - will close this option and return to the CW388 Software Window



Print Setup

This option allows for the setup of the type of printer to be used, the orientation of the printout and the size paper to be used.



Printer

The printer that is used for the majority of the printing when using the computer controller. In some cases, this may be a black and white printer.

Allows for the selection of another printer type such as a color printer for printing out color distributions.

Paper

The paper sizes available depend on the model printer that is being used. By clicking on the down arrow located to the right of the Size window a listing of the available paper sizes for the computer being used displays. The default is set to Letter 8 1/2 x 11 since most printers accommodate for this size.

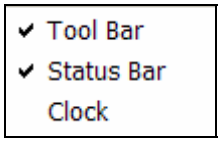
The default for this option is set to Portable Sheet Feeder however, some printers have two paper trays for printing. The source of the paper feed depends on the model of printer being used.

Orientation

The data distributions will display vertically on the paper selected or horizontally on the paper selected.

VIEW MENU

Click on View in the Main Window in order to pull down the View Menu. In the default condition, both the Tool bar, containing the icons near the top of the Main Window, and the Status Bar, located at the bottom of the Window, are activated (checked). They should remain activated.



When the user enters the Zeta mode there are a number of menus and options that change from the regular sizing software. Only those differences are listed here.

Toolbar

Increase the scattering intensity by moving the ND filter.

Decrease the scattering intensity by moving the ND filter.

Apply the E-field.

Turn OFF the E-field.

Increase the E-Field strength.

Decrease the E-Field strength.

Apply the Reference Beam.

Turn OFF the Reference Beam.

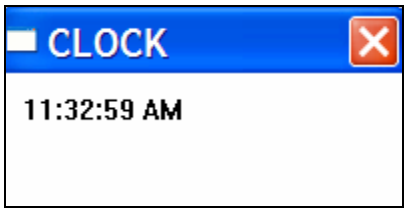
Pull sample.

Status Bar

The status bar provides pertinent information while running the Nicomp 380.

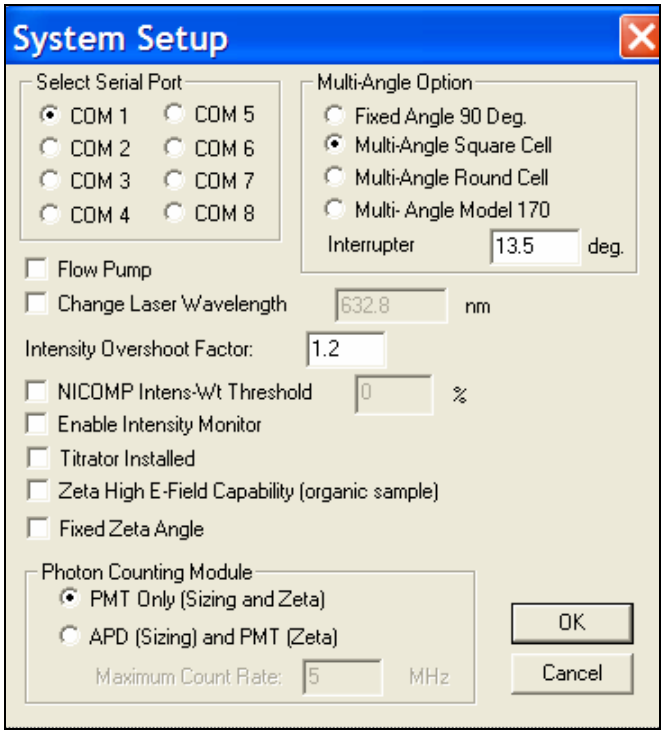
Clock

Displays the real time clock that is set up in the windows operating system of the computer. This is the clock that is used to date and time stamp the data files that are saved.



SETUP

The Setup window allows for communication to be established between the Nicomp 380/ZLS and the computer controller.



Select Serial Port

Four serial ports are provided for setting up communications between the Nicomp and the computer.

Position the cursor over the desired selection and click on the corresponding circle. A black circle will display next to the selection and the parameter will appear in the System Setup menu.

Multi-Angle Option

This parameter is used to establish the configuration for the detection of scattered light. There are four possible configurations to choose from:

Fixed Angle 90 Deg.

Selected when using the basic Nicomp in which the scattering angle is set to 90 degrees.

Multi-Angle Square Cell

Selected if the Nicomp possesses the multi-angle option (computer controlled stepper motor (0.9 deg./step) optical fiber) and a square cuvet (either normal 1-cm or miniature) is used for the sample cell. (Range 10 – 170^{o})

Multi-Angle Round Cell

Selected when the Nicomp possesses the multi-angle option and a cylindrical sample cell.

The true scattering angle is equal to the external angle of the stepper motor arm, provided the cell is highly cylindrical and well aligned – i.e. centered on the shaft of the stepper motor.

Caution: Do not use the round cell at any angle except 90o unless you are using an index matched cell.

Multi-Angle Model 170

Designed to be used with the Nicomp 170 Computing Autocorrelator. Any value for the actual scattering angle, independent of the type of scattering cell used may be entered.

Interrupter

The interrupter angle parameter is only used for the Nicomp units which include the multi-angle option.

The interrupter angle is the reference angle for the moveable arm on the stepper motor, which carries the pinhole/optical fiber receiver. When power is first applied to the Nicomp, the internal computer causes the stepper motor arm to rotate until it intersects an optical interrupter. The latter defines the reference point, or interrupter angle, which is approximately 122.4 degrees with respect to the forward direction of the laser beam (which defines the zero angle).

Caution: It may differ for other instruments. Consult PSS if you experience any problems measuring a size standard.

The stepper motor than advances in the opposite direction, at 0.9 degrees/step, until the arm reaches 90 degrees in angle. The number of steps depends on the value of the interrupter angle. Any subsequent changes in angle are made from th 90-degree "resting" angle. If the moveable arm becomes misaligned, the resting angle may differ from 90 degrees. The resulting error can easily be eliminated by resetting the value of the interrupter angle. If the resting angle is too small (e.g. 88 degrees), the interrupter angle must be decreased by the appropriate amount.

Flow Pump

The flow pump parameter indicates whether the Nicomp contains a flow pump, which is required for the Autodilution option. The flow pump may be operated manually or by automatic computer control, in Autodilution mode. Deactivate the flow pump by selecting this option.

Drop-in Cells

Use of the flow pump must be suspended when using a drop-in cell to take a measurement. If it is not, flooding in the unit will occur causing major damage to the instrument.

Change Laser Wavelength

The appropriate laser wavelength for the type of external laser being used Is entered using this option. The default wavelength is 632.8 nm which is required for the basic Nicomp with internal, 5-mW HeNe laser.

LASER	WAVELENGTH
RLD 5 MW HENE	632.5 nm
RLD 12 MW HENE	635 nm
RLD 35 MW HENE	639 nm
RLD 50 MW HENE	664 nm
RLD 100 MW HENE	664 nm
GLD 20 MW HENE	532 nm
GLD 50 MW HENE	532 nm
GLD 100 MW HENE	532 nm

Intensity Overshoot Factor

When the intensity falls to approximately the Intensity Setpoint which is set in the Conrol Menu, the pump in the Nicomp will halt, thereby stopping the flow of fresh diluent. The Intensity Overshoot Factor compensates for the variation found in the bearing of different pumps.

NICOMP Intens-Wt Threshold

This option eliminates any part of a distribution when the entire intensity contributed by that peak is less than the value specified in this item.

Enable Intensity Monitor

Provides intensity as a function of time. For kinetics studies this option allows the user to monitor and record the intensity vs time for a given solution.

Titrator Installed (optional)

There is an optional autotitrator for producing ? electronic curves in the zeta potential world. It allows for the titration of a sample with buffer and reports the values at each pH buffer point.

Zeta High E-Field Capability (organic sample)

Organic dispersions that do not conduct electricity well require the high voltage option to create an electronic field between the electrodes to monitor the zeta potential of the suspension.

Fixed Zeta Angle

The zeta interrupted comes in two “flavors”; a fixed zeta fiber at -19^{o} external and -14.7^{o} internal or a multi-angle zeta fiber which is included with the multi-angle system to perform a zeta measurement in the formal direction of + 19^{o} external and + 14.7^{o} at the internal angle.

Photon Counting Module

PMT Only (Sizing and Zeta)

APD (Sizing) and PMT (Zeta)

High gain detector offers 7 times the gain of a standard PMT for sizing small nanoparticles 0.1-10 nm or low concentration colloidal solutions.

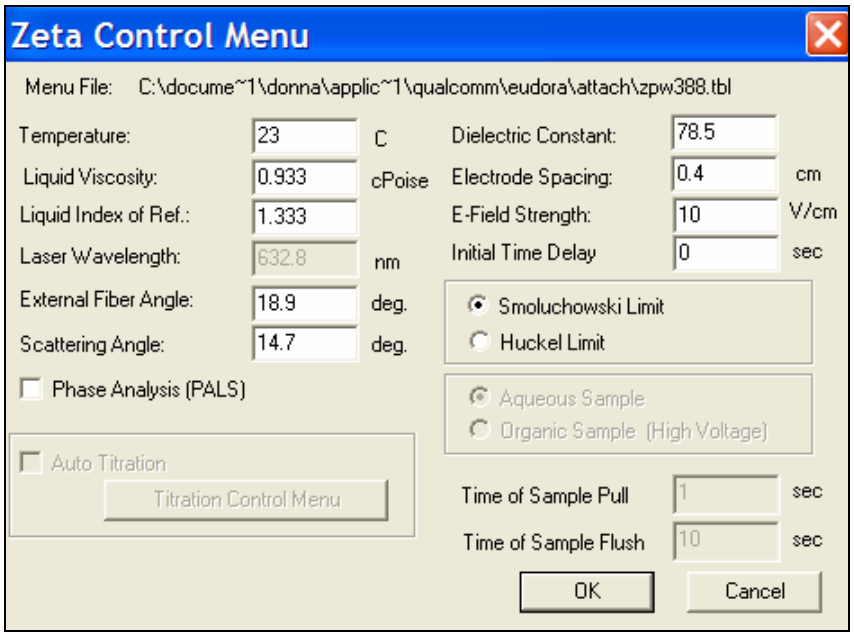
Maximum Count Rate

An APD can be damaged by exposing it to high levels of scattered light. This value automatically shuts down the APD if the value of the scattering intensity exceeds the value in the table to avoid damage. Normally this value is around 1000Khz.

ZETA POTENTIAL MENU

Control Menu	F3
Auto Print/Save Menu	F2
Set to Reference	
Read Reference File	
Mark /Unmark Sample Freq.	
Mark/Unmark Reference Freq.	
Abs/Rel Power Spectrum	Cntl+F10
Clear Data	C
Set Zeta Pot'l Min/Max	
Control Buttons	F8
To Particle Sizing	ESC
Initialize ND Filter	Alt+Ctrl+I
Titration Control menu	
Initialize Titrator	
Start Titration	
Measure pH	
Read Menu File	F7
Save Menu File	Ctrl+F7

Control Menu



Temperature

This is the temperature that the cell is set to. It can be incremented by 0.2 degree/step.

Liquid Viscosity

The viscosity of the liquid is directly related to the diffusion of particles suspended in the liquid. Using the table provided in Appendix B, enter the proper viscosity for the liquid being used.

Liquid Index of Ref. (Refraction)

The index of refraction of the liquid is entered in this field. The particles in the liquid are at such low concentrations that you don't want them to have an impact on the measurement.

Laser Wavelength

Input the proper wavelength of the laser being used. Please refer to the Wavelength table in Appendix C.

External Fiber Angle

This is the actual angle that the zeta fiber is set to + 18.9 degrees for a moving fiber and -18.9 degrees for a fixed cycle.

Scattering Angle

The internal angle taking into account the Fernell band of the glass to air interface -14.7 fixed and + 14.7 mobile fiber.

Phase Analysis (PALS)

The zeta instrument can operate in frequency of Phase analysis mode. For 99.9% of the applications

Dielectric Constant

The ability of the liquid to polarize and it change with ionic strength and pH is reported as the Dielectric Constant. The Dielectric Constant for DI water is 78.5. The Dielectric Constant is one of the variables used in the calculation of zeta from mobility.

Electrode Spacing

This value is always set as 0.4 cm. It is the physical distance between the electrodes.

E-Field Strength

The E-Field Strength can be set in the control menu but can also be adjusted using the icons to increase and decrease the E-Field during or before a measurement.

Smoluchowski Limit

Refers to a condition of high salt where there is screening and Ko = 1

Huckel Limit

Refers to a condition of a low salt where the conductivity of the solution is low and the Ko = 3/2.

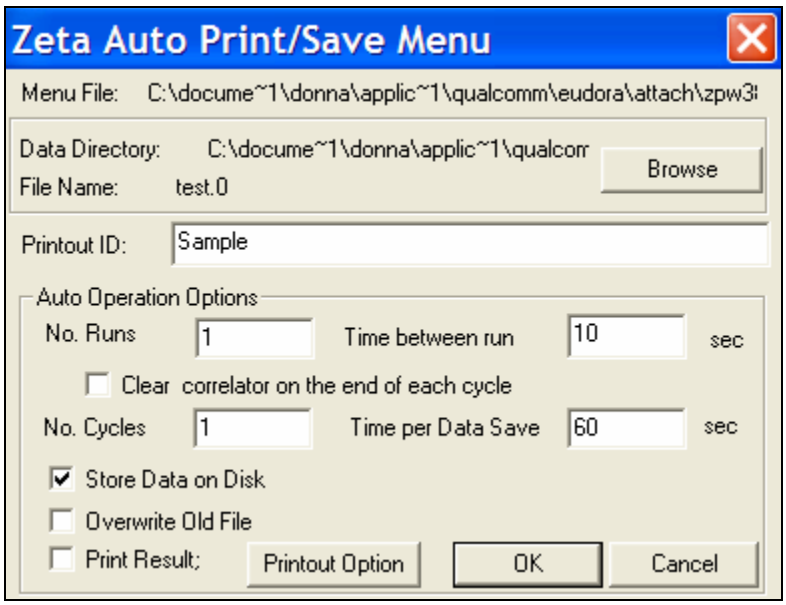
Initial Time Delay

A time delay at the start of a zeta measurement to allow the dispersion to reach equilibrium.

Time of Sample Pull and Time of Sample Flush

Refers to those systems that have the zeta flow through cell in tritration. A special option that is available as a standalone feature or as part of and autotitrator. It is the for the auto-titrator.

Auto Print/Save Menu



Data Directory

Data should never be stored to the root (C:) directory. From within the Browse function you can create a new folder to place data in. The File name will also display once defined using the Browse option.

Caution: If the directory has many levels it may cause the program to fail upon saving. Go no more than 3 levels deep in data directories when allocating a data directory.

Printout ID

Allows a maximum of 80 characters to be entered as a description. This description will appear on all printouts for the sample run.

Auto Operation Options

Allow the user to automate data taking. Each task can be done manually using the tool bar icons as well.

No. of Runs

By selecting more than 1 run the user can use the same sample for multiple measurements.

Time between run

The user may choose to let the sample rest between each run for a chosen amount of time. This can be useful in combating and Joule heating that may begin to cause convection currents within the sample cell thereby moving particulate around and out of the inspection zone.

Clear Correlator on the end of each cycle

The user may choose to clear all past information from the correlator if performing multiple measurements or allow the information to accumulate.

No. Cycles

The number of files saved to illustrate a measurement. In one run a user can set multiple save cycles.

Time per Data Save

The time of each cycle of saved data.

Example: If # of cycles = 2 and time per =30 sec. or # of cycles =1 and time per =60sec. the total measurement time for both conditions will be 60 seconds. In the first set of conditions there will be 2 files saved with increasing extension numbers each 30 seconds worth of data, in the second set only a single file containing all data for the 60 seconds.

Store Data on Disk

This option may be set to automatically save data during a sample run.

Overwrite Old File

Allows the user to overwrite old files. This option is very dangerous to use since new data can overwrite old data.

Print Result

Automatic printing at the end of a sample cycle can be performed when this option is selected.

Printout Option

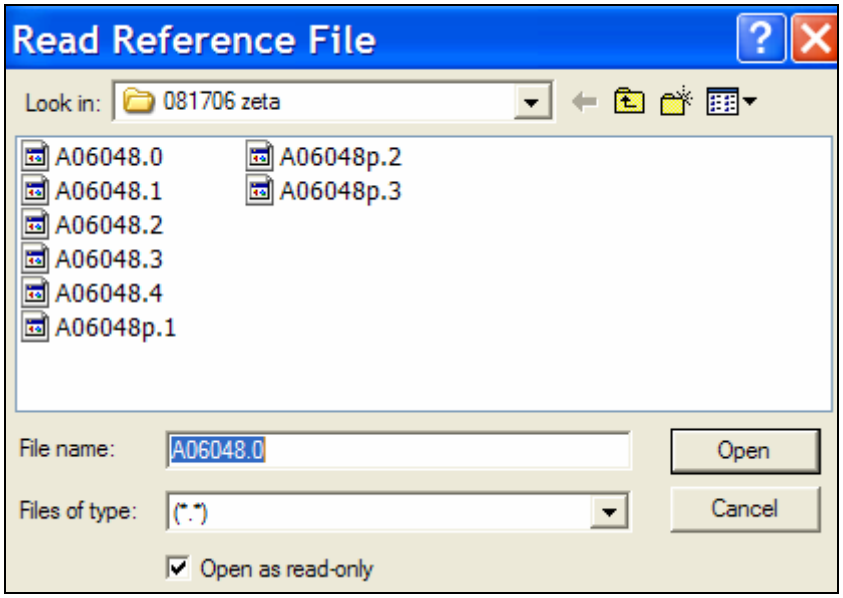
Automatic generation of the following reports may be performed provided the report type is checked. Samples of the reports can be seen in Appendix A.

Set to Reference

Sets the power spectrum as seen currently on the screen to the reference signal. The line will turn red to indicate it is now reference.

Read Reference File

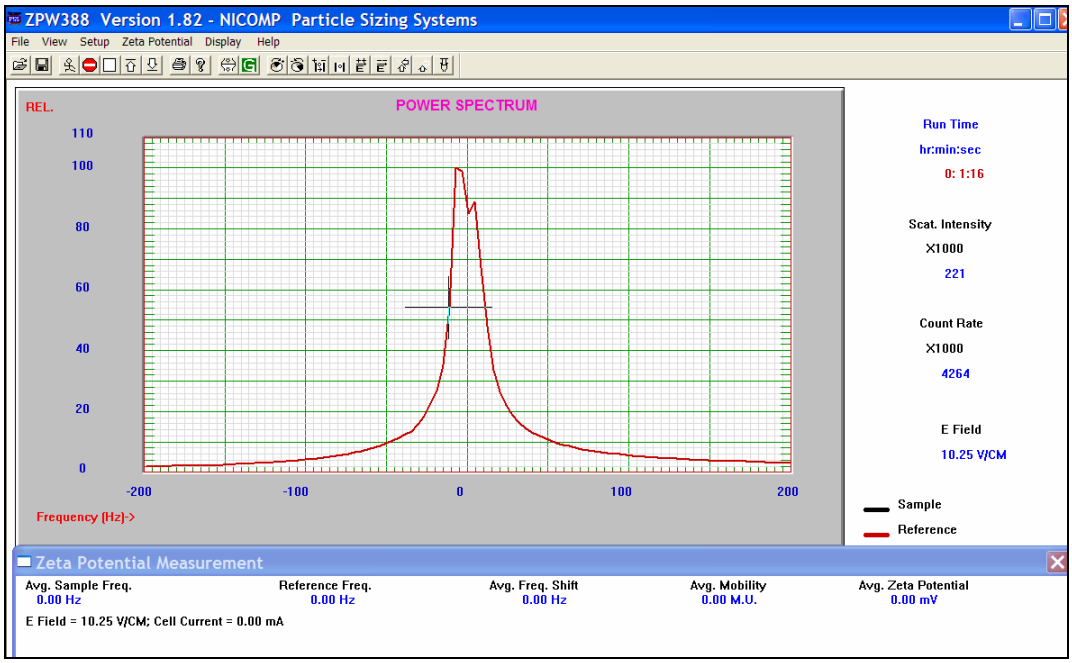
A previous stored data file may be accessed and displayed using this option.



Mark/Unmark Sample Freq.

This option can be used to manually set and obtain parameter information on a peak of the distribution.

Once selected a crosshair displays on the graph of the power spectrum. A value at any point of the sample frequency may be defined by pressing Enter. Notice that the Avg. Sample Freq. value reported on the bottom of the window will change once a new value has been selected. Also, the Power Spectrum will shift according to the new value selected. A red vertical line will display indicating the new sample frequency.



Mark/Unmark Reference Freq.

A crosshair displays on the graph of the power spectrum. The user is able to define a new position for the Reference Frequency by pressing the crosshair and then pressing Enter. The Reference Frequency will display according to the value selected.

Abs/Rel Power Spectrum

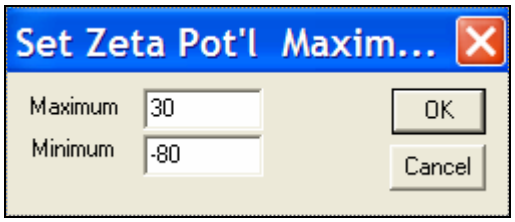
Changes the x-axis of the power spectrum graph between the reference peak being set to zero and it's absolute value.

Clear Data

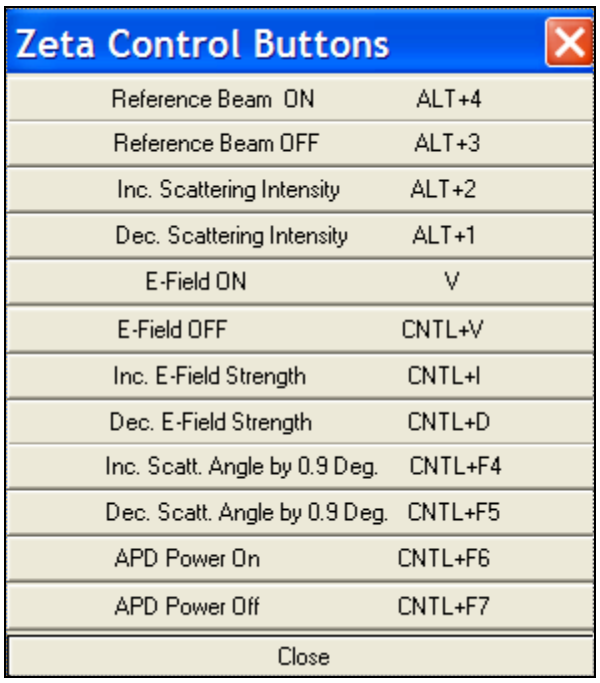
The correlator will be cleared of any accumulated data once the option is selected.

Set Zeta Pot'l Min/Max

Allows the min and max of the x-axis to be defined. This is helpful when the graph does not show the distribution adequately. For instance, the peak value is 55 and the scale displayed is 50-0. The peak is off the scale. The situation may be remedied by manually setting the x-axis of the graph.



Control Buttons


Zeta Control Buttons
Reference Beam ON ALT+4
Reference Beam OFF ALT+3
Inc. Scattering Intensity ALT+2
Dec. Scattering Intensity ALT+1
E-Field ON V
E-Field OFF CNTL+V
Inc. E-Field Strength CNTL+I
Dec. E-Field Strength CNTL+D
Inc. Scatt. Angle by 0.9 Deg. CNTL+F4
Dec. Scatt. Angle by 0.9 Deg. CNTL+F5
APD Power On CNTL+F6
APD Power Off CNTL+F7
Close

Reference Beam ON / Reference Beam OFF

Control of power to the reference beam is maintained when selecting these options.

Inc. Scattering Intensity / Dec. Scattering Intensity

The ND filter can be rotated in the desired direction to increase or decrease the scattering intensity of 120 kHz by selecting these options. This position of the ND will be stored in memory so that when the unit returns to Particle Sizing mode, the ND wheel will return to this position.

E-Field ON / E-Field OFF

Power to the E-Field is controlled using this option.

Inc. E-Field Strength / Dec. E-Field Strength

The E-Field strength is set in the Control menu but may be adjusted by increasing and decreasing the field before or during a measurement using these options.

Inc. Scatt. Angle by 0.9 Deg. / Dec. Scatt. Angle by 0.9 Deg.

The scattering angle may be changed manually using the stepper motor controlling the fiber optic. This is only to be changed when using the Particle Sizing mode of the software not the Zeta Potential option.

APD Power On / APD Power Off

The APD is used in Particle Sizing mode only and MUST not be turned on when performing a zeta measurement.

To Particle Sizing

Switches over the Particle Sizing menu to perform a particle size analysis.

Initialize ND Filter

Titration Control menu

Initialize Titrator

Start Titration

Measure pH

Read Menu File

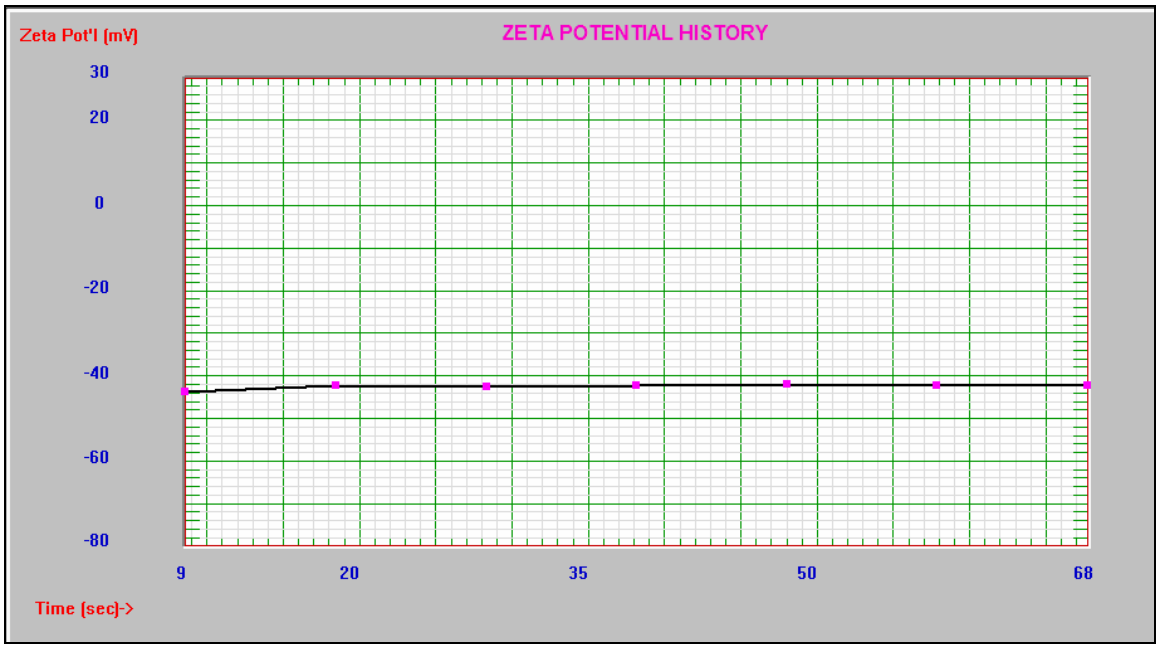
Save Menu File

DISPLAY MENU

Zeta Potential History	
Mobility History	
Phase Shift History	
Show Detail	Alt+S
Show Summary	
Zeta Potential Dist.	A
X2 Scale Change	X
pH vs. Zeta Potential	Alt+N

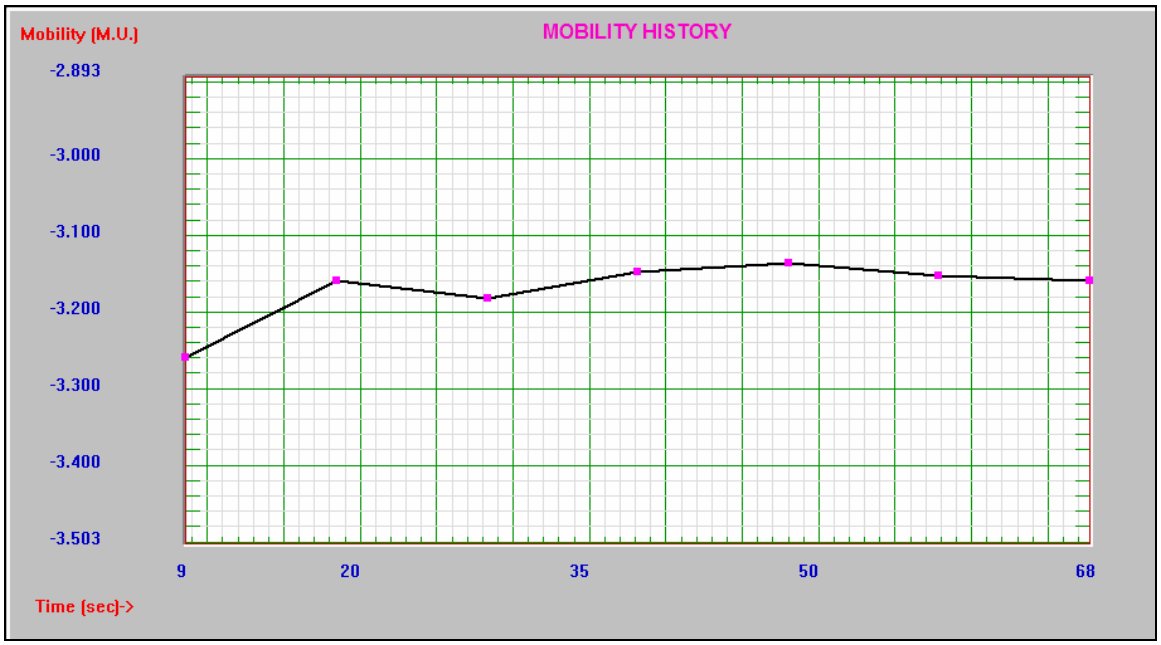
Zeta Potential History

A critical graph that provides a visual display of the stability of the zeta potential measurement over time. The numerical results may be seen in the Show Detail.



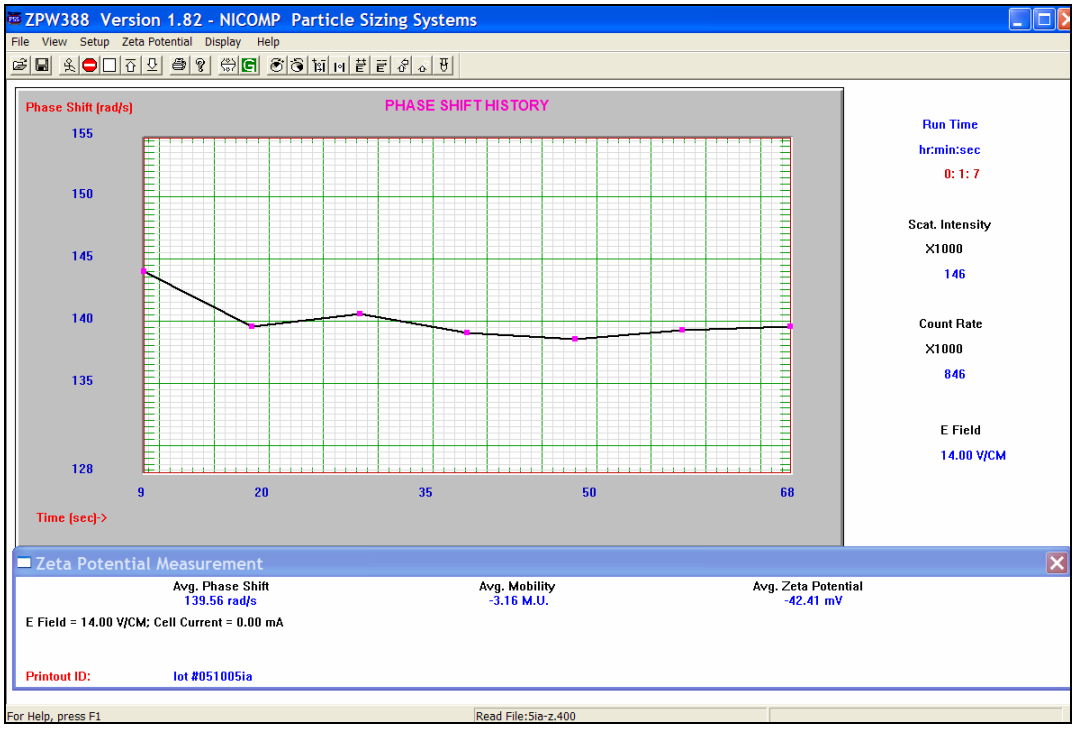
Mobility Distribution

A critical graph that provides a visual display of the mobility of the zeta potential measurement over time. The numerical results may be seen in the Show Detail.



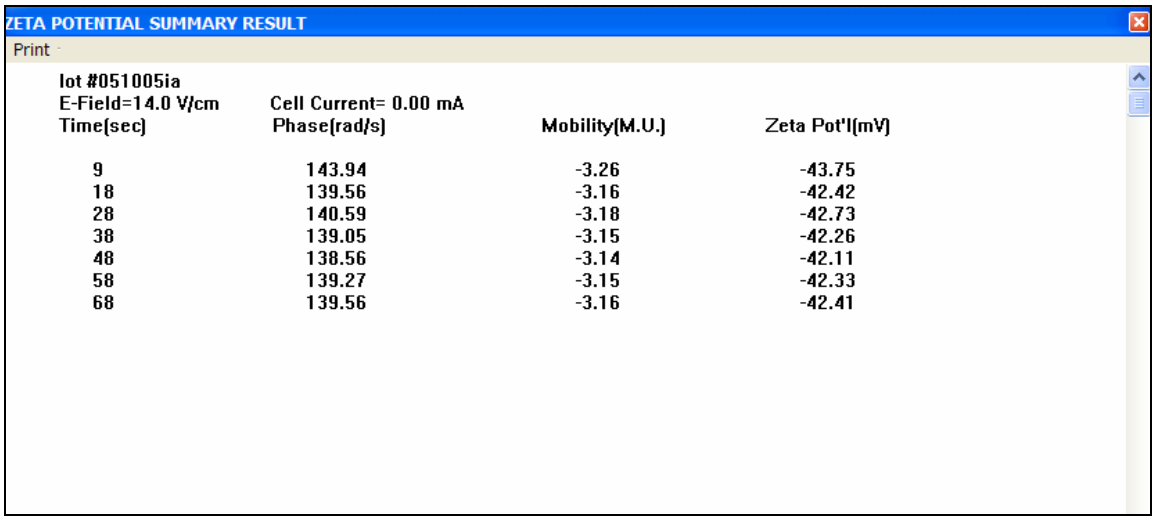
Phase Shift

A critical graph that provides a visual display of the phase shift of the zeta potential measurement over time. The numerical results may be seen in the Show Detail.



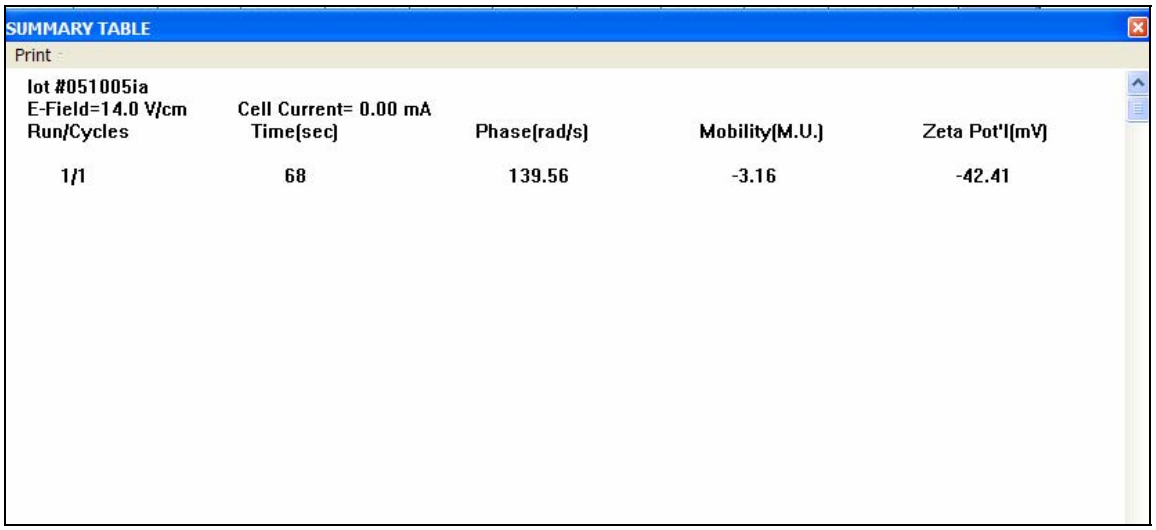
Show Detail

The Zeta Potential Summary Result window display with the following data:

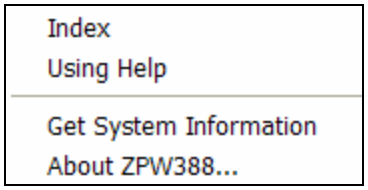


Show Summary

The Summary Table displays with the following data:



HELP



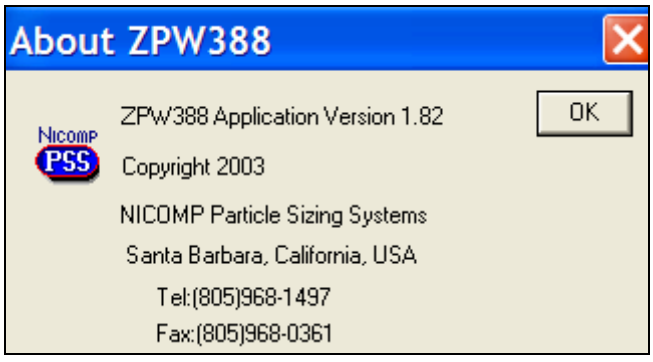
Index

The index is a listing of the older keystroke commands that have been replaced by icons, buttons and menu choices.

Using Help

Get System Information

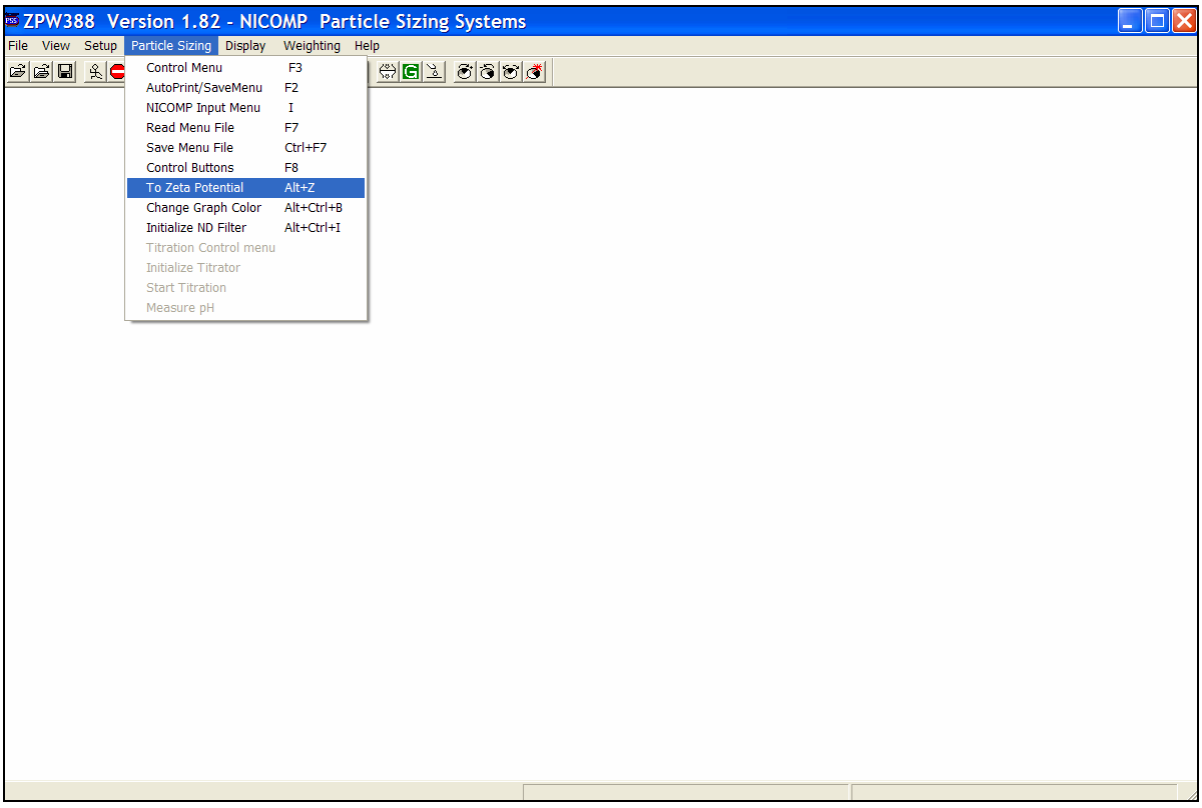
About ZPW388



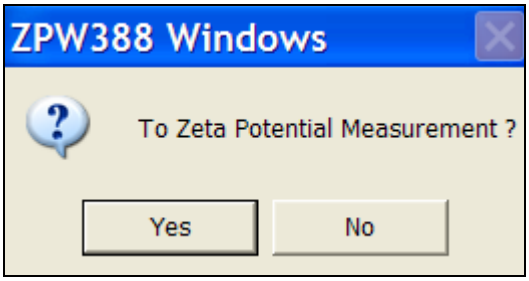
This provides some of the information required when troubleshooting the system such as software version number, etc. It also provides users with a link to the Particle Sizing Systems website which provides access to a listing of telephones numbers that can be used to call for help.

SAMPLE ANALYSIS

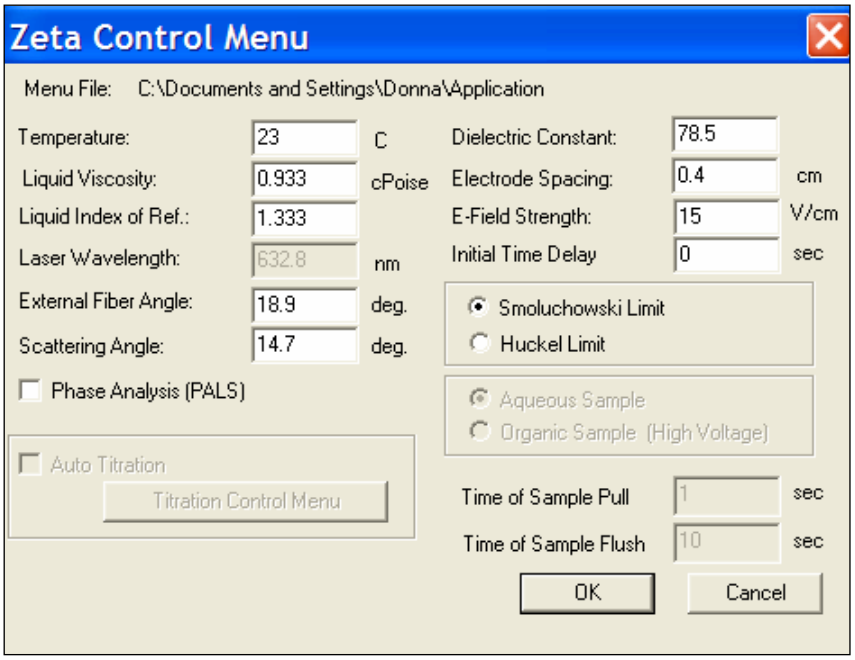
1. To transfer to Zeta Potential Software, click on Particle Sizing located on the Tool Bar, as seen on the following screen:



2. Position the highlight bar over the To Zeta Potential option and click once. The fiber optic arm can be heard to move from 90 degrees to 19 degrees. The following window will display:



The Zeta Control Menu will appear at the bottom of the screen:



The entries should read as displayed in the following table.

Temperature:	23° C
Liquid Viscosity:	0.933 cP
Liquid Index of Refraction:	1.333
Laser Wavelength:	632.8 nm
External fiber angle:	18.9 degrees
Scattering angle:	14.7 degrees
Phase Analysis (PALS)	
Dielectric constant:	78.5
Electrode spacing:	0.4 cm
E-field Strength:	15 v/cm
Initial Time Delay	0 sec
Smoluchowski Limit	
Huckel Limit	
Time of Sample Pull:	1 sec
Time of Sample Flush:	10 sec

3. Click on **OK** if the entries are correct. If they are not, position the cursor in the field(s) that need to be corrected.

4. Click once and the correct value(s).

5. Prepare the sample cuvet by filling it about 2/3 full with sample.

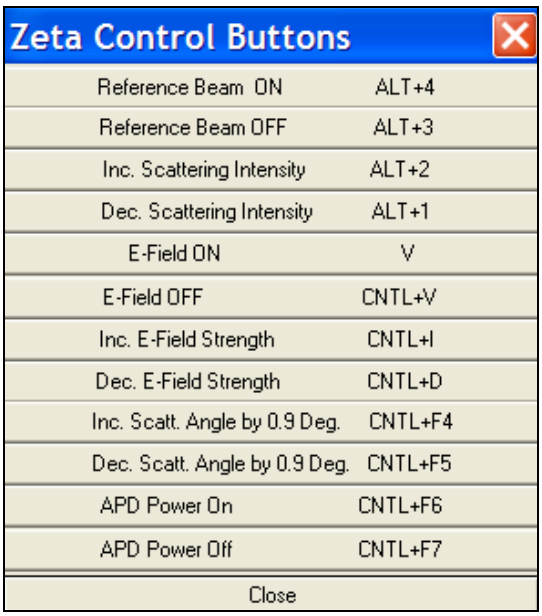
6. The liquid sample level should adjusted so that the top surface of the liquid lies a little bit below the plastic piece that holds the electrodes. This will prevent capillary "creep" of the liquid up the inside surface of the cuvet and onto the outer surface, where it can distort the optical quality of the cuvet surface. Insert the electrodes and clean the cuvet surface with a lint free wipe.

7. Insert the cuvet with electrode assembly into the sample cell holder, with the pins of the electrical connector pointed toward the back of the unit. Attach the zeta power cable to the plug.

8. Close the trap top to the unit. **Wait for 5 minutes for the sample to temperature equilibrate**. If the sample does not have a uniform temperature throughout the cuvet currents will disturb the particulate whose scattering the instrument is measuring. The most common side effect is a split peak on the frequency distribution.

9. Position the highlight bar over the Zeta Potential option and click once.

10. Select the Control Buttons option. The following window will display with a selection of buttons:


Zeta Control Buttons
Reference Beam ON ALT+4
Reference Beam OFF ALT+3
Inc. Scattering Intensity ALT+2
Dec. Scattering Intensity ALT+1
E-Field ON V
E-Field OFF CNTL+V
Inc. E-Field Strength CNTL+I
Dec. E-Field Strength CNTL+D
Inc. Scatt. Angle by 0.9 Deg. CNTL+F4
Dec. Scatt. Angle by 0.9 Deg. CNTL+F5
APD Power On CNTL+F6
APD Power Off CNTL+F7
Close

11. Position the highlight bar over the Reference Beam OFF option and click once. The motion of the shutter may be heard.

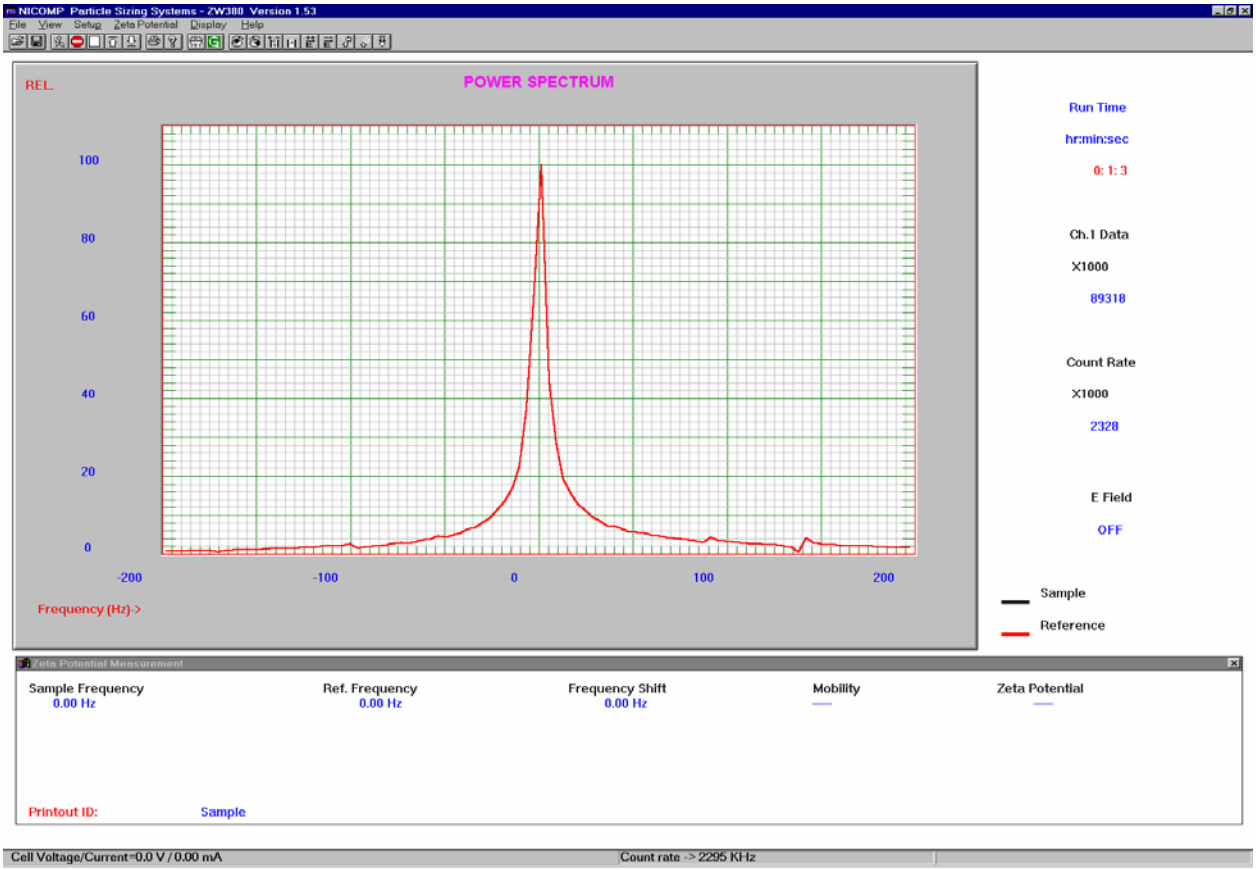
12. Adjust the scattering intensity to approximately 75-150kHz by clicking on the Inc. Scattering and Dec. Scattering Intensity buttons.

13. Click on Reference Beam ON after achieving a satisfactory scattering intensity and ensure that the reference scatter value is approximately 10-15 times of that achieved with reference beam off. If this is not achieved the sample's concentration should be altered (low scattering can mean either an over concentration or under concentrated sample, high scattering always means an over concentrated sample). This step assumes the reference beam has been aligned.

14. Click on Reference Beam OFF and click Close.

15. Press **R** to obtain a reference spectrum. The vibration of the piezoelectric can be heard. Within 20 seconds, a power spectrum will appear on the screen. It should display at 261 Hz +/-2Hz.

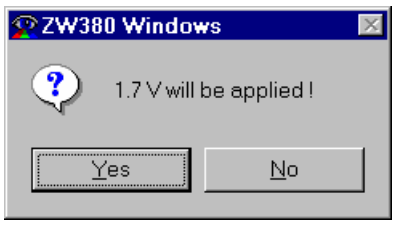
16. Continue to collect a reference signal until the 261Hz peak is the most dominant signal in the power spectrum. It should be sharp and narrow. This should take 60 seconds to achieve and should look like the below:



17. Select **Zeta Potential** on the Tool Bar.

18. Position the highlight bar over the **Set to Reference** option and click once a satisfactory reference is obtained. The spectrum will change to red.

19. Select the E-Field ON icon. A small window will display prompting if a certain voltage is appropriate:

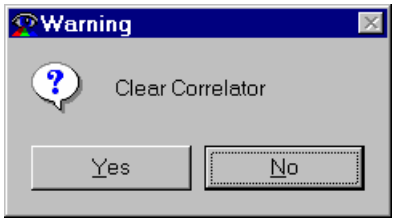


20. Select Yes if the voltage is set as desired, if not click on No and use the Increase and Decrease E-Field Strength and turn the E-Field back on.

21. Select either **Inc. E-Field Strength** or **Dec. E-Field Strength** to adjust the field. The left-most cell in the status line (bottom of the screen) indicates the voltage and current. The current should be less than 1mA. If not, decrease the E-Field strength.

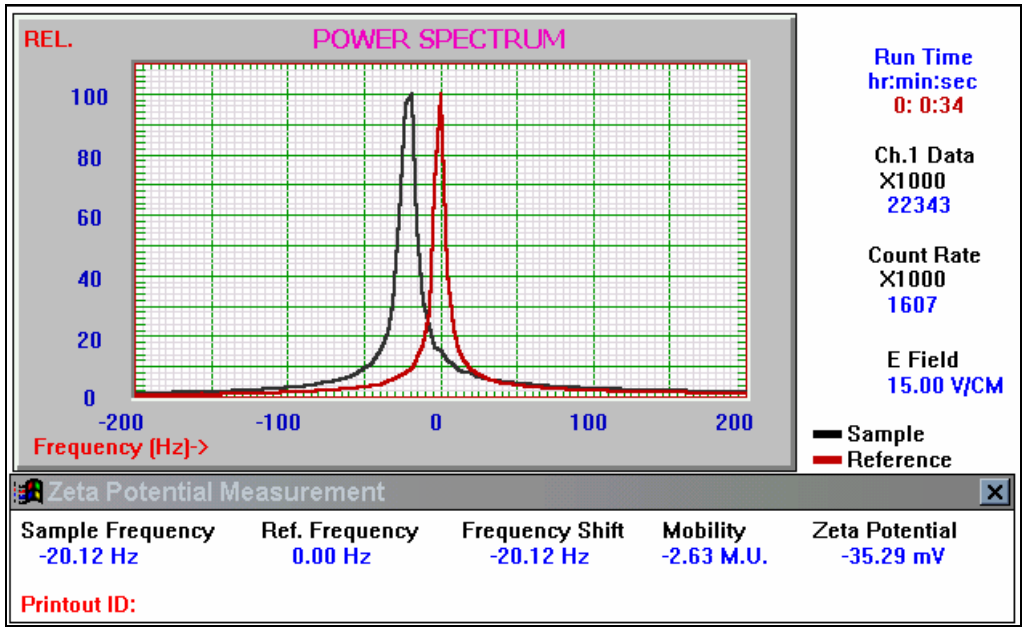
    Note: If the current is less than 1mA the user may adjust the E-Field Strength to a greater value.

22. Press C to clear the correlator. A small screen displays prompting to clear data.



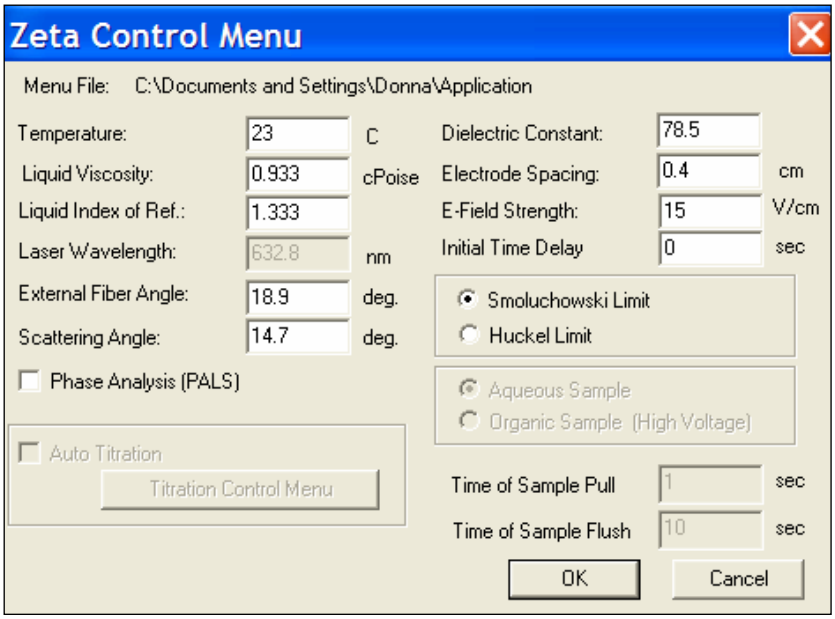
23. Select Yes. After 20 seconds, a black spectrum appears overlaid on the red power spectrum. At the bottom of the screen, a Zeta Potential Measurement window displays. It will contain the degree of frequency shift and the calculated zeta potential.

24. Select the Save Data file icon to save a data file when acceptable zeta potential information is obtained. The result may look something like the below:



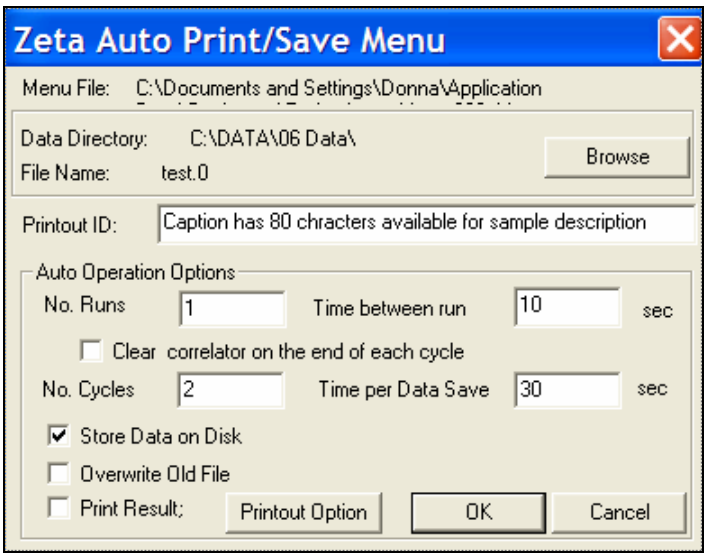
AUTOMATIC ZETA SAMPLE ANALYSIS

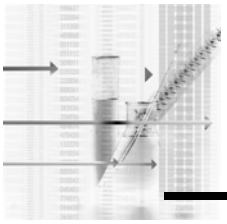
1. From the Particle Sizing Menu choose To Zeta Potential and the following screen will appear.



2. Set values as indicated in the above.

3. From the Zeta Potential Menu choose Auto Print/Save Menu and the following screen will appear.





4. Specify a place to store the data collected. Data should never be stored to the root (C:) directory. From within the Browse function you can create a new folder to place data in.

5. Printout ID allows a maximum of 80 characters to be entered as a description of the sample that will appear on all printouts.

6. Enter the number of runs in No. of Runs field. By choosing more than 1 run the same sample is used for multiple measurements.

7. Enter the Time Between Runs. The user may choose to let the sample rest between each run for a defined period of time. This can be useful in combating Joule heating that may begin to cause convection currents within the sample cell thereby moving particulate around and out of the inspection zone.

8. Indicate whether the Correlator should be cleared at the end of each cycle. The user may choose to clear all past information from the correlator if performing multiple measurements or allow the information to accumulate. This feature is useful when separating sections of data taken over time. This will allow the user to take snapshots of what happens to the sample's zeta potential over time with no bias from the past measurements.

9. Enter the No. of Cycles. This number indicates the number of files saved to illustrate a measurement.

10. Enter the Time per Data Save. The time of each cycle of saved data.

    Example: If the # of cycles = 2 and time per = 30 sec. or # of cycles = 1 and time per = 60sec. the total measurement time for both conditions will be 60 seconds. In the first set of conditions there will be 2 files saved with increasing extension numbers each 30 seconds worth of data, in the second set only a single file containing all data for the 60 seconds.

11. Chose to store the data on disk or to automatically print after each run. Overwriting the file name is not recommended for systems that utilize a current PC since each file is just a few Kilobytes of information and there is room to save many files.

12. Select OK and prepare the sample.

13. Prepare the sample cuvet by filling it about 2/3 full with sample.

14. The liquid sample level should be adjusted so that the top surface of the liquid lies a little bit below the plastic piece that holds the electrodes. This will prevent capillary “creep” of the liquid up the inside surface of the cuvet and onto the outer surface, where it can distort the optical quality of the cuvet surface. Clean the cuvet surface with a lint free wipe.

15. Insert the cuvet with electrode assembly into the sample cell holder, with the pins of the electrical connector pointed toward the back of the unit. Attach the zeta power cable to the plug.

16. Close the trap top to the unit. **Wait for 5 minutes for the sample to temperature equilibrate**. If the sample does not have a uniform temperature throughout the cuvet currents will disturb the particulate whose scattering the instrument is measuring. The most common side effect is a split peak on the frequency distribution.

17. Select the start measurement icon.

18. The instrument will adjust the ND filter until scattering intensities are optimized for the zeta measurement. A small window will appear stating this action is taking place. If the error “sample too concentrated please dilute” appears, dilute the sample by 50% and attempt the measurement again. If the error persists try initializing the ND filter. If the zero point on the ND filter has been lost by the software the full range of the filter is no longer available and the instrument assumes that the reason the scattering intensities are too large is due to the sample concentration.

19. A window will appear stating that data collection has begun with the E-Field off. The reference signal is being measured and stored. This step takes approximately 1 min.

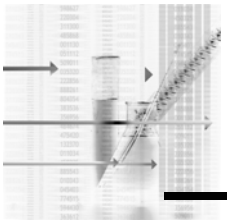
20. The small window towards the bottom of the screen will show the number of cycles and runs to be completed. It will also display a count down of the time remaining between runs.

21. After the measurement is completed it may be necessary to turn off the E-Field manually. Check the E-Field status on the screen before removing the electrodes.

22. Files may be read by clicking on the Read Data icon and searching out the file of interest.

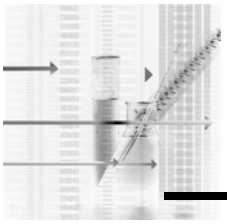
23. If multiple samples are to be run one may use the same reference for each and a window at the start of any subsequent measurement will ask “Do you want to measure reference Y/N”.

Zeta Sample Analysis (Titration)



Zeta Sample Analysis (Titration)

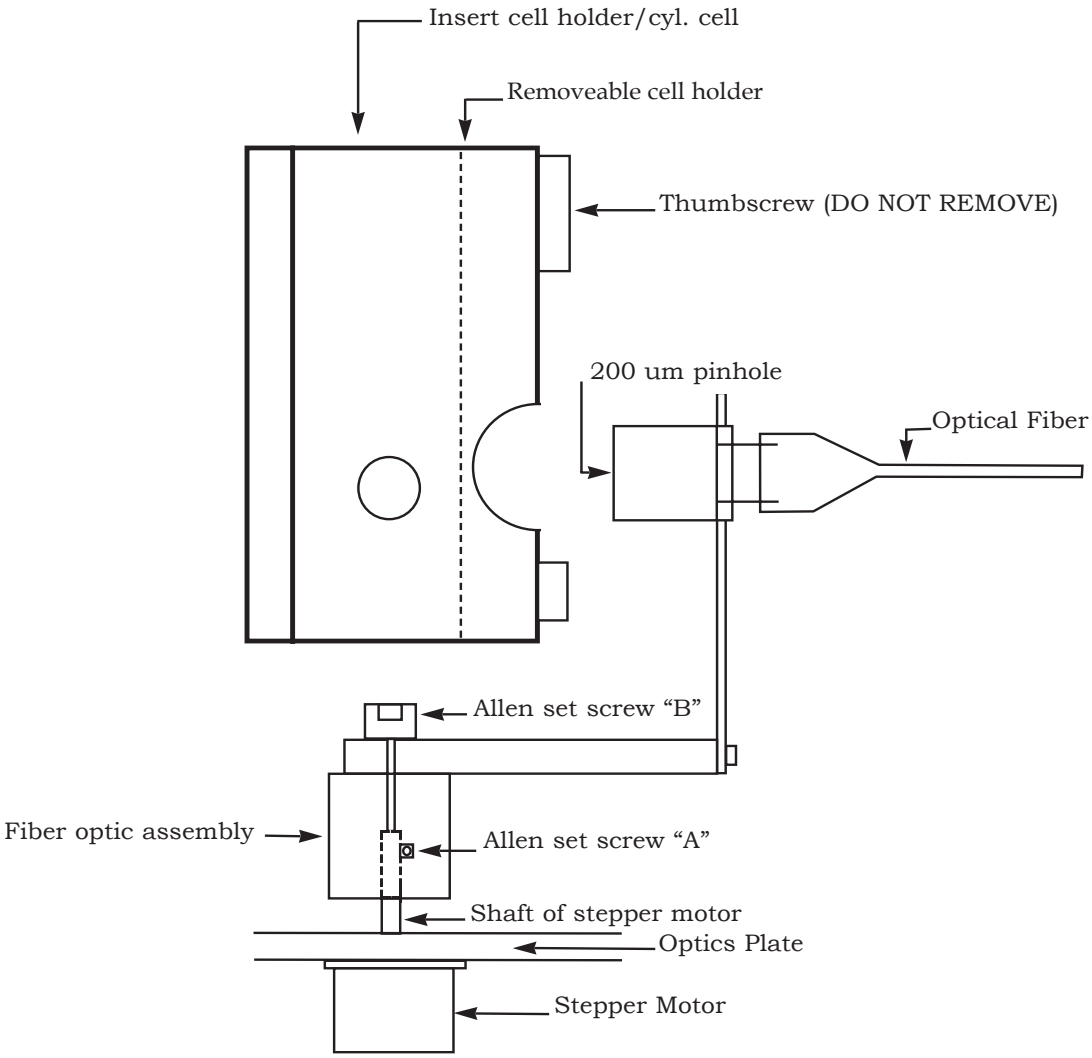
Zeta Sample Analysis (Auto- Titration)

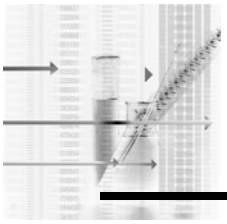


ALIGNMENT

Below is a sketch of the fiber-optic assembly and cell holder of the multi-angle system, utilized in the Nicomp instruments, as viewed from the FRONT of the unit:

Fiber Optic and Cell Holder Assembly





ALIGNMENT

The procedure for aligning the optical fiber arm/assembly follows:

1. Insert a dilute sample solution (e.g. 260 nm latex in water) into the cell holder, using a disposable plastic (1cm) cuvet. Adjust the sample concentration so that the scattering intensity is approximately 200-400KHz.

2. From the Control Menu request an external angle of 60 degrees. The moveable fiber-optic arm will then go to that angle, bringing into better view set screw "A", which fastens the fiber optic assembly onto the shaft of the stepper motor.

3. Move the arm back to 90 degrees (approx.). The exact angle is UNIMPORTANT.

4. Using a 3/32" Allen wrench, loosen set screw "A" SLIGHTLY, so that the arm assembly is able to move up and down on the motor shaft, but not too loosely.

5. Remove the sample cuvet from the cell holder.

6. Insert the same Allen wrench into the space normally occupied by the cuvet and secure it into the cap of Allen screw “B”.

7. Rotate the screw by approximately one (1) full turn COUNTER-CLOCKWISE. This will cause the screw to retract approx. 0.25" from the end of the motor shaft.

8. Press firmly down on the Allen screw/arm assembly, so that the end of the retracted screw will come into firm contact with the end of the motor shaft.

   Note: If set screw "A" is still too tight, the arm assembly may not be free to move downward with respect to the motor shaft. Test for this by trying to turn screw "B" CLOCKWISE by a fraction of turn, to determine whether the crew encounters resistance, indicating that it is in contact with the end of the motor shaft. If necessary, set screw "A" should be loosened a bit more.

9. Reinsert the sample cuvet into the cell holder and note carefully the new scattering intensity. The value should be lower than the value noted previously, because of the substantial misalignment just effected. The optical fiber should now be too low with respect to the "line" source of light scattering within the sample cuvet. In order to improve the alignment, the arm must therefore be RAISED.

10. Remove the sample cuvet and reinsert the Allen wrench into the cap of screw "B". The short arm at the end of the wrench can be used like the hand of a clock.

11. Align the wrench at the start so that its "hand" points away from the front panel, i.e. approx. at 12:00 on a clock dial.

12. Rotate the wrench CLOCKWISE by 1/4 turn, so that the hand points to approx. 3:00. This has the effect of RAISING the fiber/arm by approx. 3/40", or 0.075".

13. Reinsert the sample in the cell holder and check the scattering intensity. It should either be unchanged or somewhat higher than the previous value.

14. Repeat the operation above as many times as necessary, in order to maximize the scattered light signal. If the value reaches or exceeds 1000 kHz, further dilute the sample suspension, to avoid saturating the PMT signal or damaging the detector. Of course, one typically cannot locate the position that produces the maximum intensity unless the maximum has been exceeded, with screw "B" rotated too far in the clockwise direction. Hence, one must then rotate the screw COUNTER-CLOCKWISE by approx. 1/4 to 1/3 turn, so that one can go backwards and repeat the earlier operation of carefully RAISING the fiber/arm, in order to locate with precision the alignment which produces the maximum scattered intensity.

15. Finally, set screw "A" must be tightened, to secure the arm assembly onto the motor shaft. One should verify that the scattering intensity remains approximately the same after this screw has been tightened.

    CAUTION: *Set screw "A" must NOT be tightened too hard, to avoid damage to either the screw or the arm assembly.*

MAINTENANCE

If electrodes appear to have noticeable tarnish then they will have to be polished.

1. Cut a strip of 600-grit wet/dry emery paper (≈4" x ¾").
2. Adhere (with a glue stick) the strip of emery paper to a similar sized metal or wooden strip (e.g. a tongue depressor).
3. Wet the emery paper and electrode pair with clean water.
4. Holding the electrode in one hand and the emery paper in the other, rub each surface of the electrode pair below the White Delrin across the emery paper using a back and forth motion. Continue until the tarnish is removed.
5. Cut a strip of 1200-grit wet/dry emery paper (≈4" x ¾").
6. Adhere (with glue stick) the strip of emery paper to a similar sized metal or wooden strip (e.g. a tongue depressor).
7. Wet the emery paper and electrode pair with clean water.
8. Holding the electrode in one hand and the emery paper in the other, rub each surface of the electrode pair below the Delrin across the emery paper using a back and forth motion.
9. Continue until a smooth polish is achieved.

DISPOSABLE CUVETS:

The supplier for the disposable cuvets is Fischer Scientific.
The part number is 14-386-21.

DETAIL

Particle Sizing Systems, Inc.

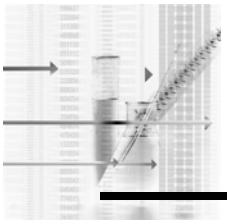
Santa Barbara, Calif., USA

lot # 051005I

Data File: C:\Work in Progress\Nicomp ZLS Manual\030806 380ZLS\test499.400 13:7:9 11/2/2006

E-Field=14.0 V/cm Cell Current= 0.00 mA

Freq. Shift(Hz)	Rel. Intensity	Mobility(M.U.)	Zeta Pot'l(mV)
-20.53	1.54	2.92	39.21
-18.31	1.75	2.61	34.97
-16.09	2.00	2.29	30.73
-13.87	2.31	1.97	26.50
-11.66	2.70	1.66	22.26
-9.44	3.20	1.34	18.02
-7.22	3.85	1.03	13.78
-5.00	4.71	0.71	9.54
-2.78	5.88	0.40	5.31
-0.56	7.55	0.08	1.07
1.66	10.00	-0.24	-3.17
3.88	13.79	-0.55	-7.41
6.10	20.00	-0.87	-11.65
8.32	30.77	-1.18	-15.88
10.54	50.00	-1.50	-20.12
12.76	80.00	-1.81	-24.36
14.98	100.00	-2.13	-28.60
17.20	80.00	-2.45	-32.84
19.41	50.00	-2.76	-37.07
21.63	30.77	-3.08	-41.31
23.85	20.00	-3.39	-45.55
26.07	13.79	-3.71	-49.79
28.29	10.00	-4.02	-54.03
30.51	7.55	-4.34	-58.26
32.73	5.88	-4.66	-62.50
34.95	4.71	-4.97	-66.74
37.17	3.85	-5.29	-70.98
39.39	3.20	-5.60	-75.22
41.61	2.70	-5.92	-79.45
43.83	2.31	-6.23	-83.69
46.05	2.00	-6.55	-87.93
48.27	1.75	-6.87	-92.17



APPENDIX A

SUMMARY RESULT (PHASE MODE ONLY)

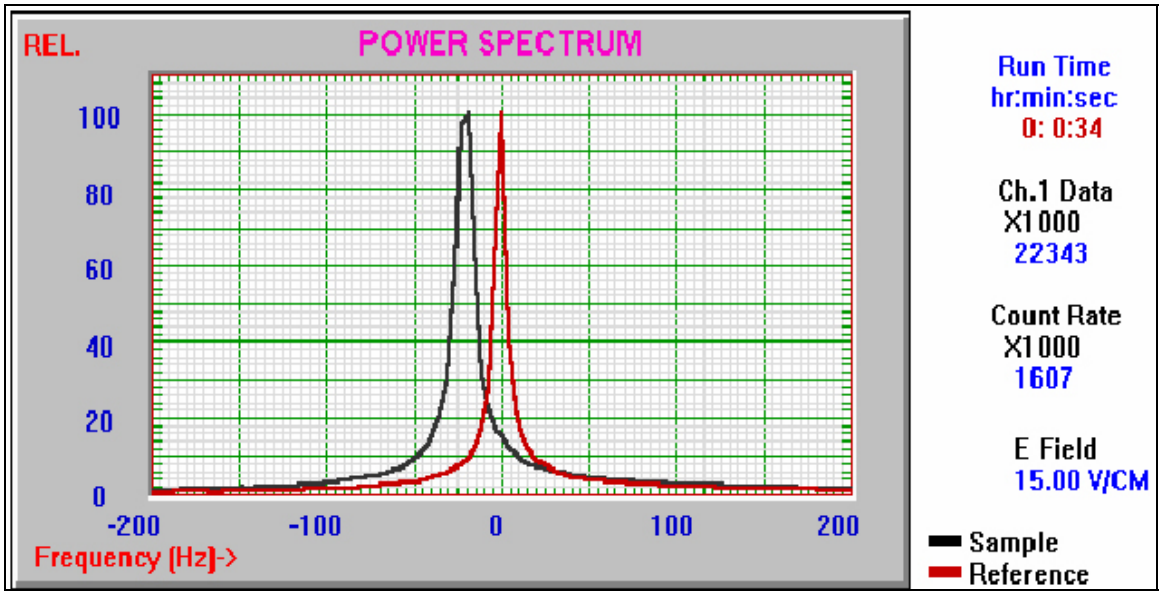
TIME HISTORY (FREQUENCY MODE ONLY)

Particle Sizing Systems, Inc.
Santa Barbara, Calif., USA

lot # 051005I
Data File: C:\Work in Progress\Nicomp ZLS Manual\030806 380ZLS\test499.400 13:7:9 11/2/2006

Sample Frequency:	275.26 Hz
Reference Frequency:	260.29 Hz
Cell Current:	0.00 mA
Frequency Shift:	14.98 Hz
Avg. Mobility:	-2.13 M.U.
Half-Width Mobility Dist.	-----
Avg. Zeta Potential:	-28.60 mV
Half-Width Zeta Pot'l Dist.	-----
Sample Temperature:	23 C
Liquid Viscosity:	0.933 cPoise
Index of Refraction:	1.333
Dielectric Constant:	78.500
Laser Wavelength:	639.0 nm
Scattering Angle:	-14.1 deg.
E-Field Strength:	14.00 V/CM
Channel Width:	500.0 uSec.
Run Time:	00:01:35

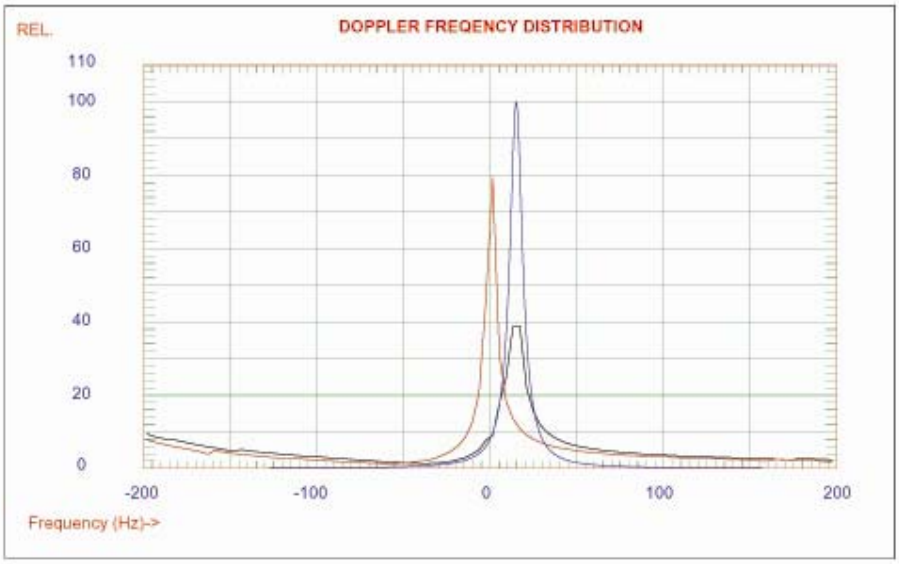
POWER SPECTRUM (FREQUENCY MODE ONLY)



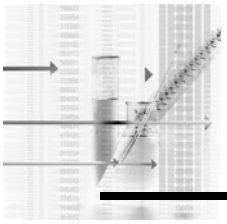
DOPPLER FREQUENCY DISTRIBUTION (FREQUENCY MODE ONLY)

Particle Sizing Systems, Inc.
Santa Barbara, Calif., USA

lot # 051005I
Data File: C:\Work in Progress\Nicomp ZLS Manual\030806 380ZLS\test499.400 13:7:9 11/2/2006



Sample Frequency:	275.26 Hz
Reference Frequency:	260.29 Hz
Cell Current:	0.00 mA
Frequency Shift:	14.98 Hz
Avg. Mobility:	-2.13 M.U.
Half-Width Mobility Dist.	-----
Avg. Zeta Potential:	-28.60 mV
Half-Width Zeta Pot'l Dist.	-----
Sample Temperature:	23 C
Liquid Viscosity:	0.933 cPoise
Index of Refraction:	1.333
Dielectric Constant:	78.500
Laser Wavelength:	639.0 nm
Scattering Angle:	-14.1 deg.
E-Field Strength:	14.00 V/CM
Channel Width:	500.0 uSec.
Run Time:	00:01:35

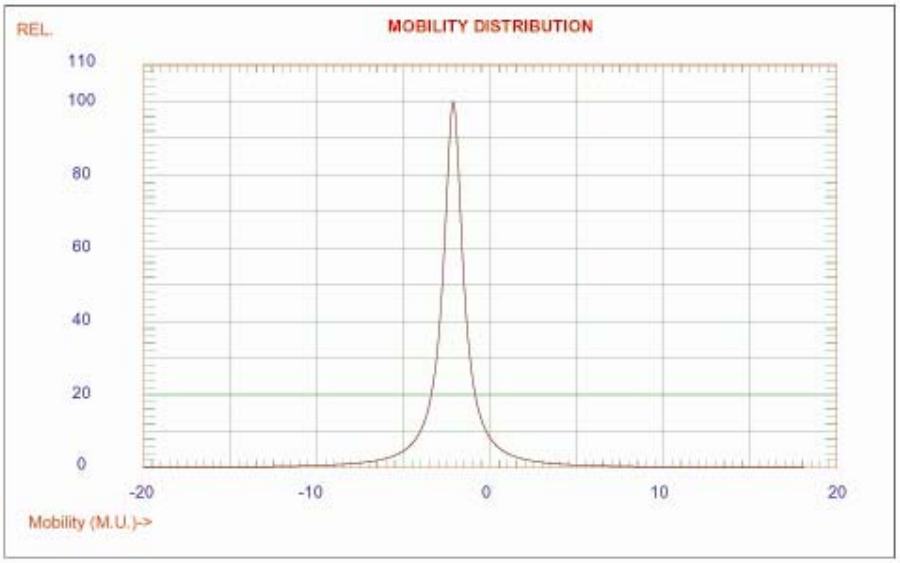


APPENDIX A

MOBILITY DISTRIBUTION (FREQUENCY MODE ONLY)

Particle Sizing Systems, Inc.
Santa Barbara, Calif., USA

lot # 051005I
Data File: C:\Work in Progress\Nicomp ZLS Manual\030806 380ZLS\test499.400 13:7:9 11/2/2006



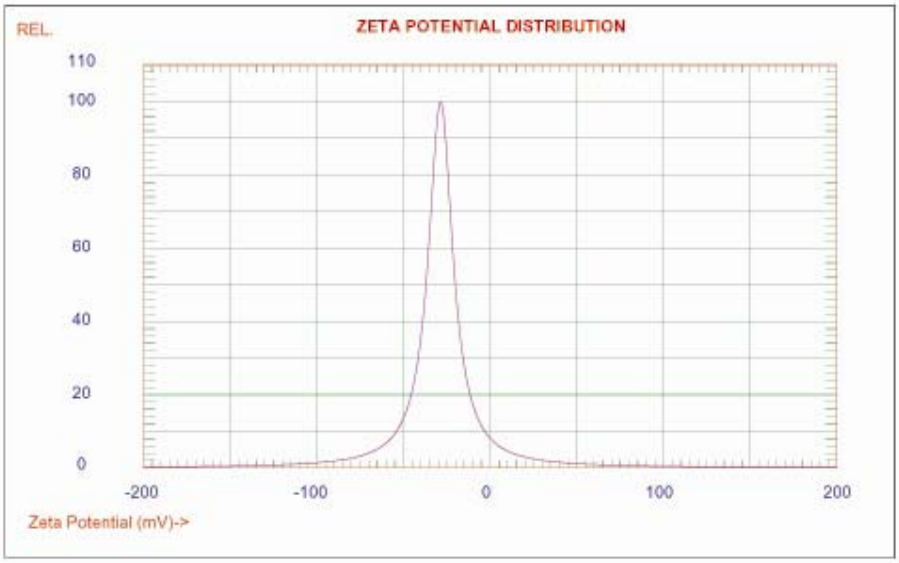
Sample Reference

Sample Frequency:	275.26 Hz
Reference Frequency:	260.29 Hz
Cell Current:	0.00 mA
Frequency Shift:	14.98 Hz
Avg. Mobility:	-2.13 M.U.
Half-Width Mobility Dist.	-----
Avg. Zeta Potential:	-28.60 mV
Half-Width Zeta Pot'l Dist.	-----
Sample Temperature:	23 C
Liquid Viscosity:	0.933 cPoise
Index of Refraction:	1.333
Dielectric Constant:	78.500
Laser Wavelength:	639.0 nm
Scattering Angle:	-14.1 deg.
E-Field Strength:	14.00 V/CM
Channel Width:	500.0 uSec.
Run Time:	00:01:35

ZETA POTENTIAL DISTRIBUTION

Particle Sizing Systems, Inc.
Santa Barbara, Calif., USA

lot # 051005I
Data File: C:\Work in Progress\Nicomp ZLS Manual\030806 380ZLS\test499.400 13:7:9 11/2/2006

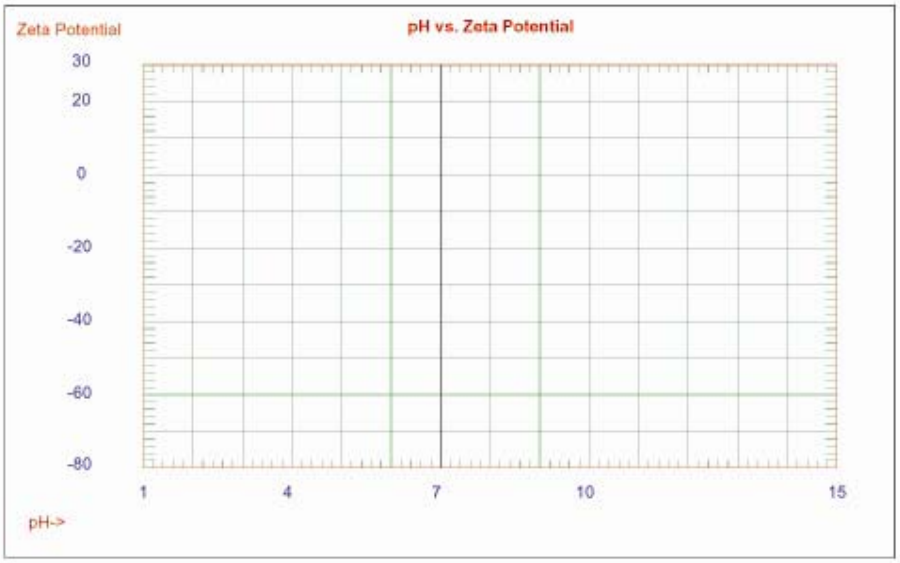


Sample Frequency:	275.26 Hz
Reference Frequency:	260.29 Hz
Cell Current:	0.00 mA
Frequency Shift:	14.98 Hz
Avg. Mobility:	-2.13 M.U.
Half-Width Mobility Dist.	-----
Avg. Zeta Potential:	-28.60 mV
Half-Width Zeta Pot'l Dist.	-----
Sample Temperature:	23 C
Liquid Viscosity:	0.933 cPoise
Index of Refraction:	1.333
Dielectric Constant:	78.500
Laser Wavelength:	639.0 nm
Scattering Angle:	-14.1 deg.
E-Field Strength:	14.00 V/CM
Channel Width:	500.0 uSec.
Run Time:	00:01:35

PH VS ZETA POTENTIAL DISTRIBUTION

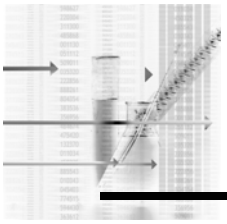
Particle Sizing Systems, Inc.
Santa Barbara, Calif., USA

lot # 051005I
Data File: C:\Work in Progress\Nicomp ZLS Manual\030806 380ZLS\test499.400 13:7:9 11/2/2006



Sample Frequency:	275.26 Hz
Reference Frequency:	260.29 Hz
Cell Current:	0.00 mA
Frequency Shift:	14.98 Hz
Avg. Mobility:	-2.13 M.U.
Half-Width Mobility Dist.	-----
Avg. Zeta Potential:	-28.60 mV
Half-Width Zeta Pot'l Dist.	-----
Sample Temperature:	23 C
Liquid Viscosity:	0.933 cPoise
Index of Refraction:	1.333
Dielectric Constant:	78.500
Laser Wavelength:	639.0 nm
Scattering Angle:	-14.1 deg.
E-Field Strength:	14.00 V/CM
Channel Width:	500.0 uSec.
Run Time:	00:01:35

ZETA POTENTIAL HISTORY (PHASE MODE ONLY)



APPENDIX A

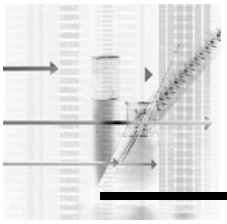
MOBILITY HISTORY (PHASE MODE ONLY)

APPENDIX A

PHASE SHIFT HISTORY (PHASE MODE ONLY)

APPENDIX B

SOLVENT	TEMP (C)	VISCOSITY (cpoise)	INDEX REFRACTION
Acetaldehyde	10	0.256	1.332
	20	0.220	1.332
Acetic Acid	15	1.31	1.380
	25	1.16	1.380
	41	1.00	1.380
	59	0.70	1.380
Acetone	15	0.337	1.357
	25	0.316	1.357
	41	0.280	1.357
Acetonitrile	15	0.375	1.346
	25	0.345	1.346
	30	0.325	1.346
n-Amyl acetate	11	1.58	1.400
	45	0.805	1.400
n-Amyl alcohol	15	4.65	1.410
	30	2.99	1.410
n-Amyl ether	15	1.188	1.410
Aniline	15	5.31	1.583
	25	3.71	1.583
	35	2.71	1.583
Benzaldehyde	25	1.39	1.544
Benzene	20	0.652	1.498
	30	0.564	1.498
	40	0.503	1.498
Benzonitrile	15	1.45	1.526
	25	1.24	1.526
	30	1.11	1.526
Benzyl Alcohol	20	5.80	1.538
	30	4.65	1.538
Benzyl amine	25	1.59	1.540
Bromoform	15	2.152	1.587
	25	1.89	1.587

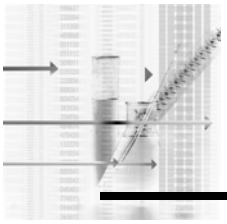


APPENDIX B

SOLVENT	TEMP (C)	VISCOSITY (cpoise)	INDEX REFRACTION
n-Butyl acetate	20	.732	1.372
	40	.563	1.372
n-Butyl alcohol	20	2.948	1.400
	30	2.3	1.400
	40	1.782	1.400
Carbon disulfide	20	0.363	1.628
	40	0.330	1.628
Carbon tetrachloride	15	1.038	1.459
	20	0.969	1.459
	30	0.843	1.459
	40	0.739	1.459
Chlorobenzene	15	0.900	1.523
	20	0.799	1.523
	40	0.631	1.523
Chloroform	20	0.58	1.444
	30	0.514	1.444
	39	0.500	1.444
Cyclohexane	15	1.06	1.426
	30	0.82	1.426
Cyclohexanol	20	68.0	1.456
	30	41.1	1.456
Cyclohexanone	15	2.45	1.450
	30	1.80	1.450
Cyclohexene	13.5	0.696	1.445
	20	0.66	1.445
Cyclopentane	13.5	0.493	1.404
n-Decane	20	0.920	1.409
	25	0.853	1.409
N,N-Dimehtylformamide (="DMF")	20	0.84	1.427
	25	0.80	1.427

APPENDIX B

SOLVENT	TEMP (C)	VISCOSITY (cpoise)	INDEX REFRACTION
Dimehtylaniline	20	1.41	1.558
	30	1.17	1.558
	40	1.04	1.558
n-Dodecane	25	1.35	1.415
Ethyl Acetate	15	0.473	1.380
	20	0.455	1.380
	25	0.441	1.380
	30	0.400	1.380
Ethyl alcohol	20	1.200	1.359
(=Ethanol)	30	1.003	1.359
	40	0.834	1.359
Ethyl benzene	15	0.697	1.495
	30	0.581	1.495
Ethyl bromide	15	0.418	1.424
	20	0.402	1.424
	30	0.348	1.424
Ethyl ether	20	0.233	1.352
	25	0.222	1.352
	40	0.197	1.325
Ethyl formate	15	0.419	1.361
	30	0.358	1.361
Ethylene bromide	17	1.950	1.538
	20	1.721	1.538
Ethylene dichloride	15	0.887	1.445
	30	0.730	1.445
Ethylene glycol	20	19.90	1.431
	30	13.35	1.431
	40	9.13	1.431
	60	4.95	1.431
Formamide	25	3.30	1.446
Formic acid	20	1.804	1.371
	30	1.465	1.371
	40	1.219	1.371



APPENDIX B

SOLVENT	TEMP (C)	VISCOSITY (cpoise)	INDEX REFRACTION
n-Heptane	20	0.409	1.388
	25	0.386	1.388
	40	0.341	1.388
n-Hexadecane	20	3.45	1.433
n-Hexane	20	0.326	1.375
	23	0.3068	1.375
	25	0.294	1.375
	40	0.271	1.375
Isobutyl alcohol	15	4.703	1.397
	30	2.876	1.397
Isopentane	20	0.223	1.355
Isopropyl alcohol	15	2.86	1.385
	30	1.77	1.385
Methyl acetate	20	0.381	1.380
	40	0.320	1.380
Methyl alcohol	20	0.597	1.326
(= Methanol)	25	0.547	1.326
	30	0.510	1.326
	40	0.456	1.326
Methyl ethyl ketone	15	0.423	1.379
(= "MEK")	30	0.365	1.379
Methyl formate	15	0.360	1.346
	25	0.328	1.346
Methylene dichloride	15	0.449	1.424
	30	0.393	1.424
Nitrobenzene	15	2.24	1.550
	20	2.03	1.550
Nitromethane	25	0.620	1.380
o-Nitrotoluene	20	2.37	1.547
	30	1.91	1.547
	40	1.63	1.547

APPENDIX B

SOLVENT	TEMP (C)	VISCOSITY (cpoise)	INDEX REFRACTION
m-Nitrotoluene	20	2.33	1.545
	30	1.77	1.545
	40	1.60	1.545
p-Nitrotoluene	60	1.204	1.533
n-Octane	20	0.542	1.395
	40	0.433	1.395
Pentane	20	0.240	1.357
Propyl acetate	20	0.59	1.382
	40	0.44	1.382
n-Propyl alcohol	20	2.256	1.385
	30	1.72	1.385
	40	1.405	1.385
1,1,2,2-Tetrachloroethan	15	1.844	1.494
Toluene	20	0.590	1.494
	30	0.526	1.494
	40	0.471	1.494
Trichlorethane	20	1.20	1.438
o-Xylene	16	0.876	1.506
	20	0.810	1.506
	40	0.627	1.506
m-Xylene	15	0.650	1.495
	20	0.620	1.495
	40	0.497	1.495
p-Xylene	16	0.696	1.493
	20	0.648	1.493
	40	0.513	1.493

APPENDIX C

LASER WAVELENGTHS

The appropriate laser wavelength for the type of external laser being used is displayed in this table.

LASER	WAVELENGTH
RLD 5 MW HENE	632.5 nm
RLD 12 MW HENE	635 nm
RLD 35 MW HENE	639 nm
RLD 50 MW HENE	664 nm
RLD 100 MW HENE	664 nm
GLD 20 MW HENE	532 nm
GLD 50 MW HENE	532 nm
GLD 100 MW HENE	532 nm